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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 69 Number 3

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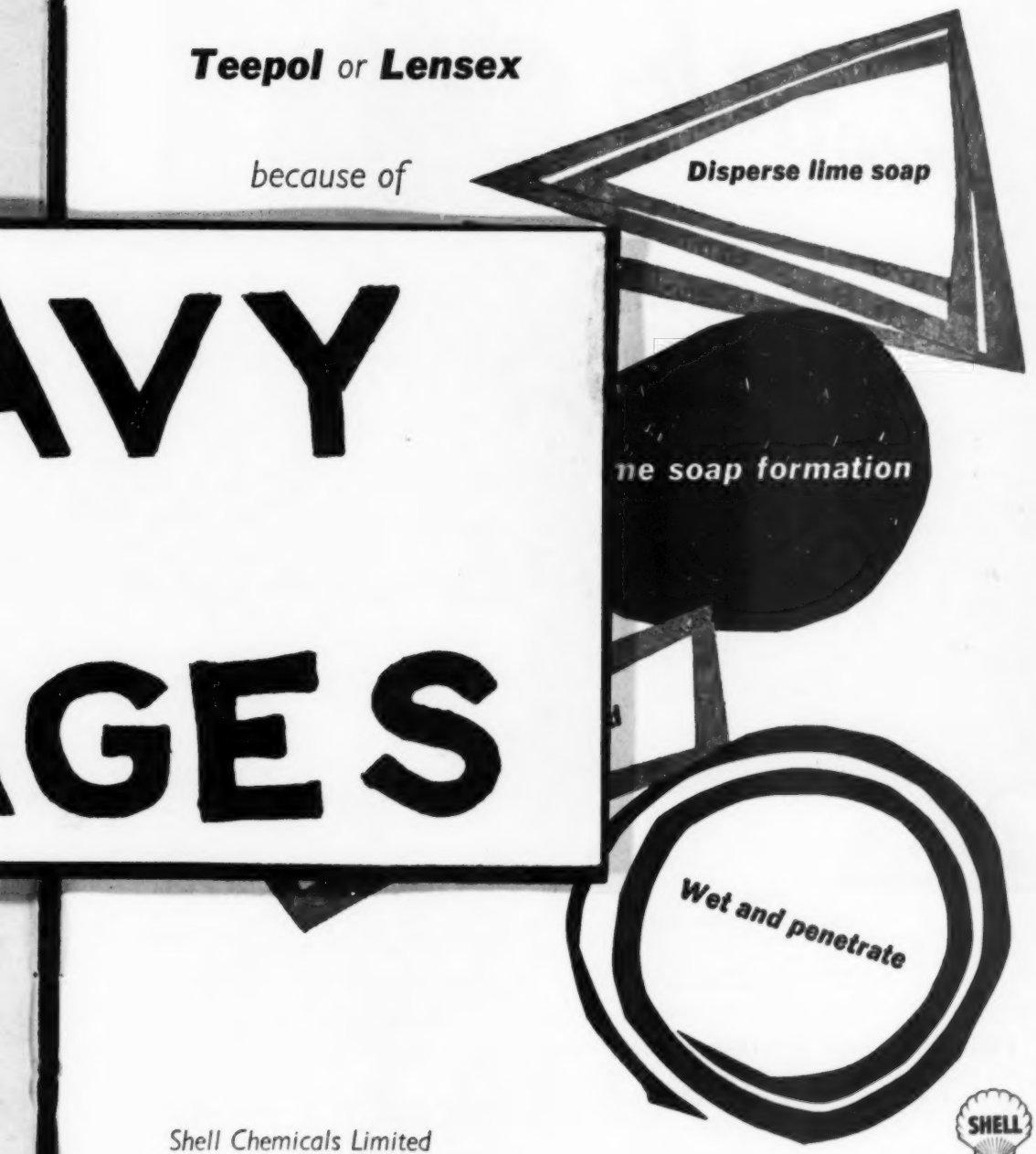

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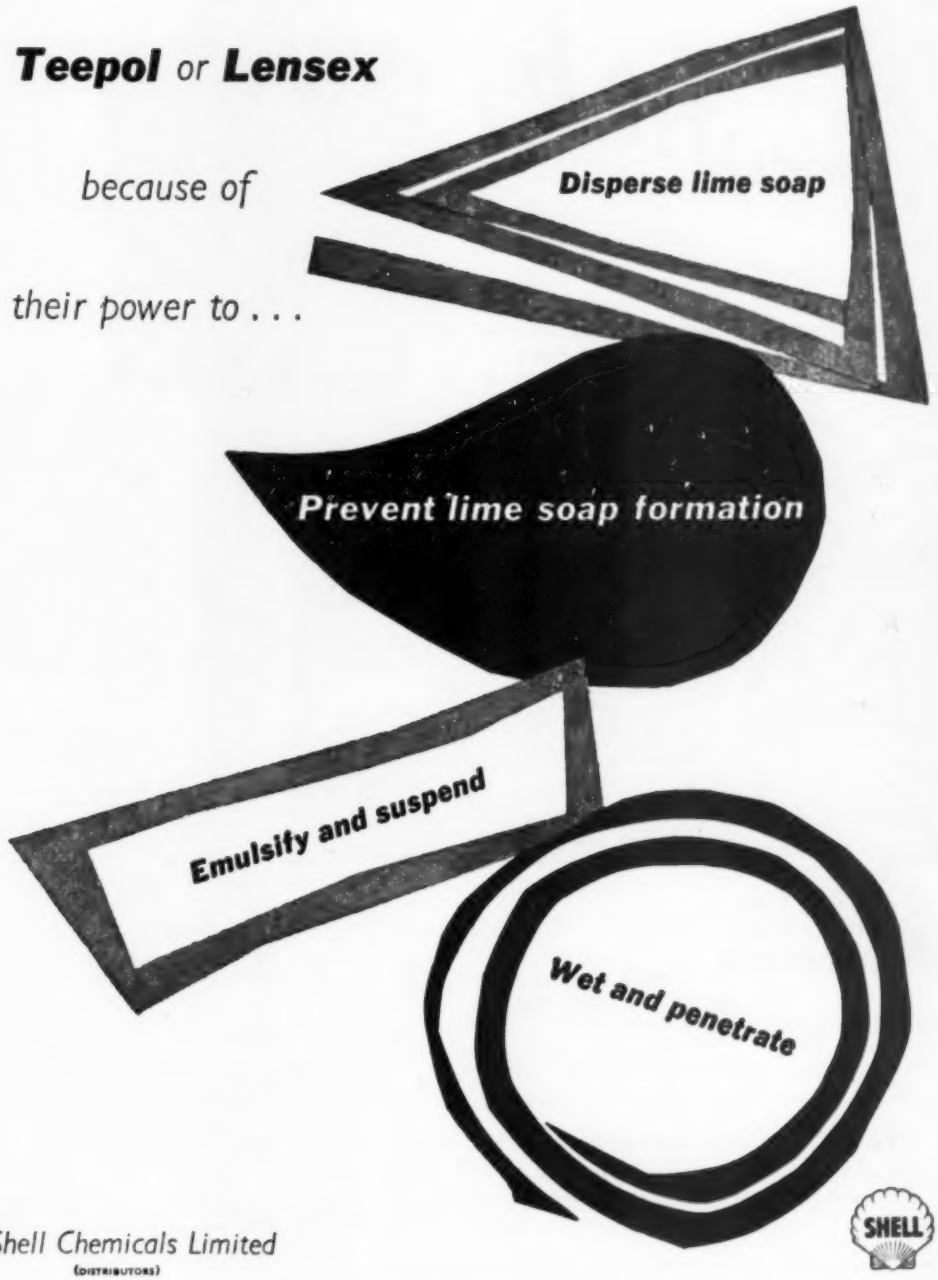

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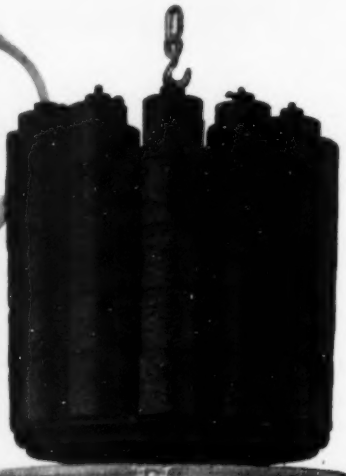

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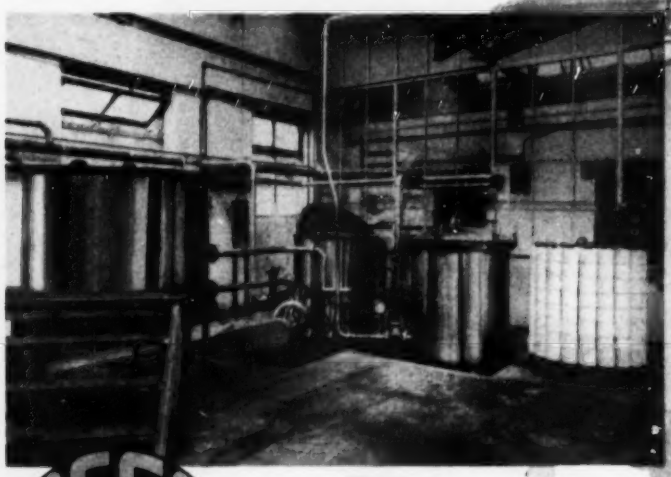

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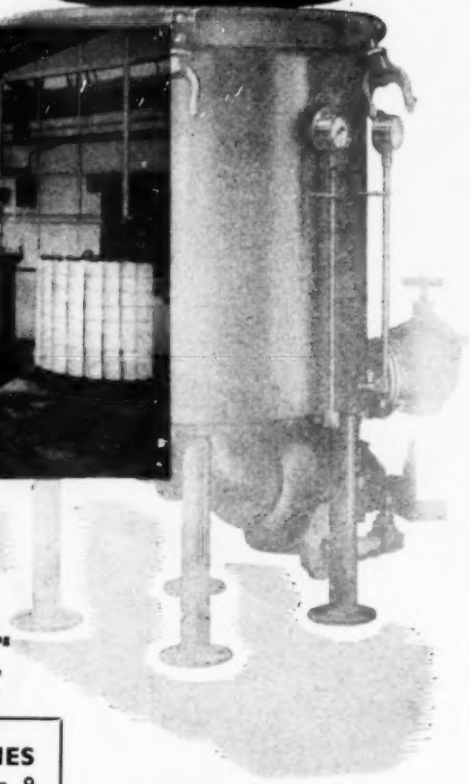

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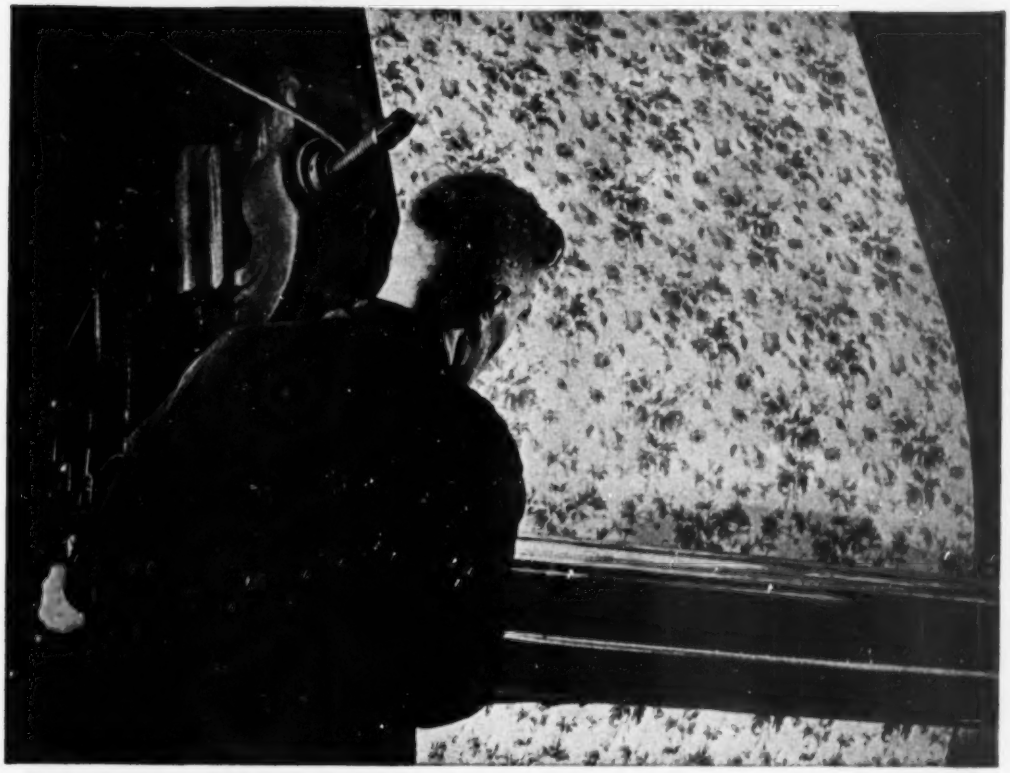

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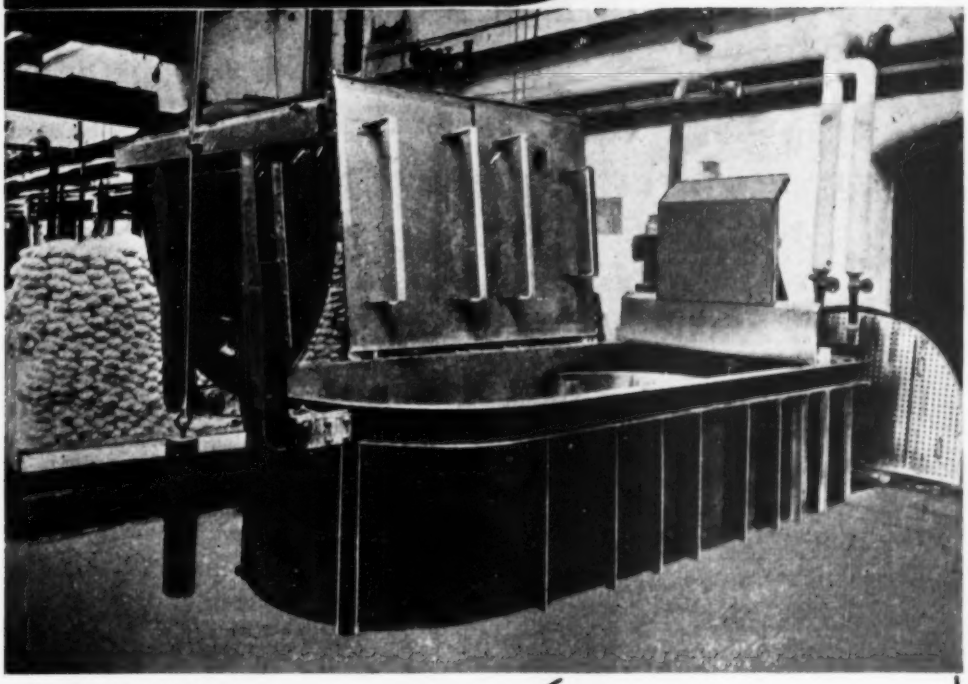

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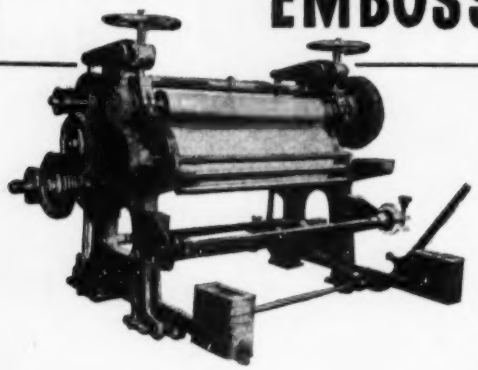

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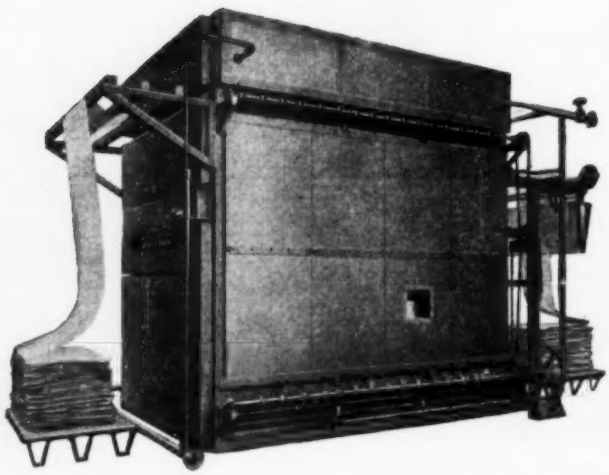

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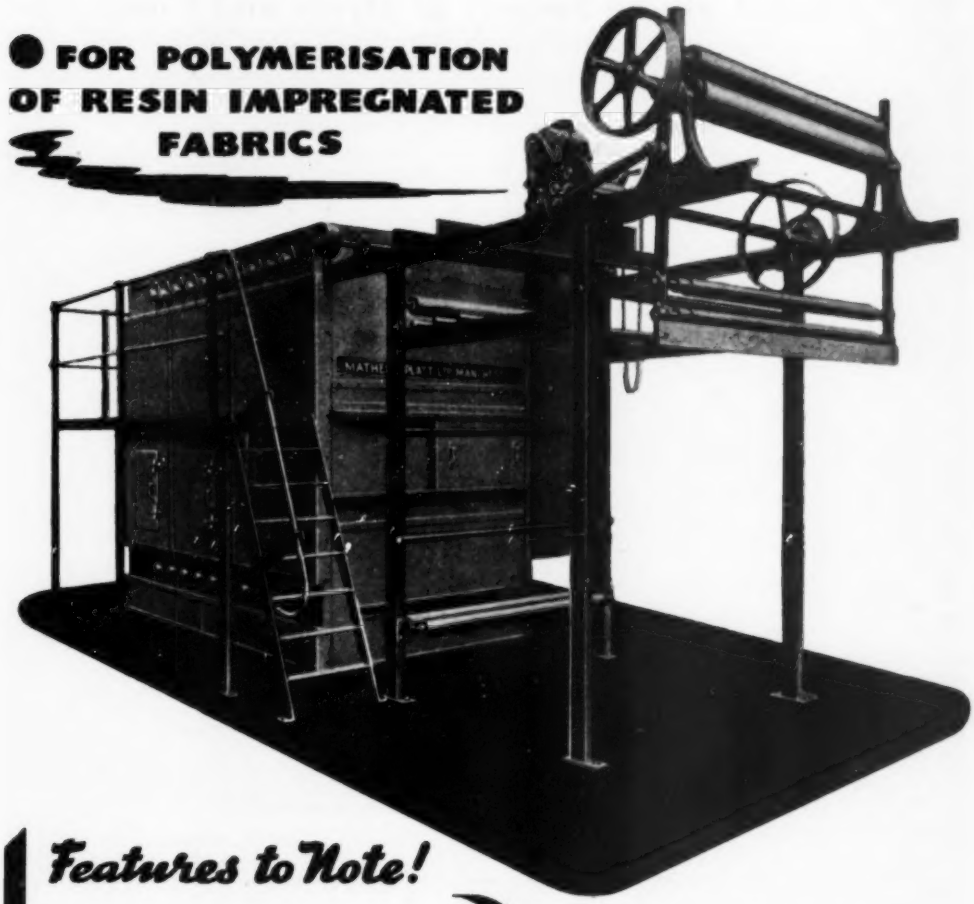

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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1953 and pages 237–240 of the July 1952 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURE

Fluorescent Dyes and the Application of Fluorescence to Textiles — *T. Thorne Baker*

COMMUNICATIONS

Zacharie Roussin — *R. Brightman*

Studies in the Fundamental Processes of Textile Printing. V— The Transfer of Disperse and Water-soluble Dyes to Cellulose Acetate during Steaming — *E. H. Daruwalla and H. A. Turner*

An Electron-microscope Examination of Wool— The Fraction resistant to Peracetic Acid–Ammonium Hydroxide Treatment — *R. L. Elliott and B. Manogue*

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Forthcoming Meetings of the Society

Friday—27th March 1953 at 7-15 p.m.

THIRD GEORGE DOUGLAS LECTURE Dr. T. Holbro *The Search for New Dyes in Relation to Modern Developments in the Textile Field* The University, Leeds

Thursday—9th April 1953 at 7-30 p.m.

TENTH MERCER LECTURE J. R. Blockey, M.Sc., F.R.I.C., and D. H. Tuck, A.L.C. *The Coloration of Leather* Royal Institution, Albemarle Street, London W.1

Friday—10th April 1953

ANNUAL GENERAL MEETING and DINNER of the Society May Fair Hotel, London W.1

MANCHESTER SECTION

Unless otherwise stated meetings are held in the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m.

1953	
Friday 17th April	ANNUAL GENERAL MEETING followed by a lecture—Professor W. Bradley. *Experiments on the Chemistry of Anthraquinone Derivatives*

NORTHERN IRELAND SECTION

1953	
Saturday 28th March	ANNUAL GENERAL MEETING and DINNER

MIDLANDS SECTION

Except when otherwise stated, Lecture Meetings commence at 7 p.m.

1953	
Wednesday 22nd April	ANNUAL GENERAL MEETING OF THE SECTION. Gas Board Theatre, Nottingham

Towards the end of the session Dr. H. White (Textile Research Institute, Princeton, U.S.A.) will lecture in Nottingham. Particulars later

HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Huddersfield, at 7.30 p.m., unless otherwise stated

1953	
Tuesday 31st March *Note altered date*	ANNUAL GENERAL MEETING followed by a lecture—Dr. J. F. Gaunt. *A Study of the Afterchrome Process of Wool Dyeing*

WEST RIDING SECTION

All meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1953	
Thursday 26th March	ANNUAL GENERAL MEETING

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THE JOURNAL

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Volume 69 Number 3 MARCH 1953 *Issued Monthly*

Proceedings of the Society

Defective Colour Vision

F. JORDINSON

Meeting of the Huddersfield Section held at Field's Café, Huddersfield, on 19th February 1952, Mr. R. L. Robinson in the chair

WHITE AND COLOURED LIGHT

When white light is passed through a glass prism, it is resolved into light of various wavelengths, which the normal eye sees as the colours of the spectrum. Collectively these colours give to the eye the impression of white, as may be shown by the rotation of a suitably coloured spinning top.

The balance of the spectrum colours is destroyed when one or more of them is removed from the "white mixture", and the eye then receives a sensation of colour. A ripe strawberry is red because it absorbs all wavelengths except red, which is reflected to the eye. Similarly, a red solution absorbs the blue-green wavelengths.

MECHANISM OF COLOUR VISION

In the retina are great numbers of sense-cells of two different kinds, known from the shapes of their ends as rods and cones.

RODS are mainly concerned with simple light perception and are especially useful at low intensities. In moonlight they enable distances to be judged and shapes to be distinguished but not colour. The periphery of the retina, which consists mainly of rods, therefore has the greatest sensitivity to very dim light, and a faint star is best viewed sideways, i.e. out of the corners of the eye.

CONES are used for acute vision in bright light and for colour perception. They are particularly numerous at the yellow spot (*macula lutea*), which occupies a small central patch in the retina in each eye; elsewhere there are far more rods than cones. This yellow spot in the retina actually contains only cones, and its centre, the *fovea*, is the point of most accurate vision.

The yellow pigment in the macula gradually thickens as a person grows older; also the lens of the eye gradually turns yellow with advancing years. In the case of an aged person, these changes cause much of the blue light to be cut off, whereas with a child nearly all the blue light is transmitted. This has an important bearing on the colour vision of older as compared with younger people, and the matter will be referred to later in this paper.

A pigment known as *visual purple* is present in the rod cells of the retina but not in the cones. It plays an important part in twilight vision, for it is bleached by bright light but is slowly re-formed in dim light. A person who passes suddenly from bright into dim light cannot see at all at first, but visual purple begins to re-form almost at once, and after a few minutes he can see fairly well. Visual purple absorbs greenish-yellow light most strongly and is most readily bleached by light of this colour.

There is a close relationship between visual purple and vitamin A. Lack of this vitamin prevents the rebuilding of visual purple which normally takes place in darkness, and thus gives rise to night-blindness. The exact chemical composition of visual purple is not known, but it appears to be a compound of a protein and a substance closely related to vitamin A*. When visual purple (obtained from the retina of a frog) is exposed to light, another pigment, called *visual yellow*, is formed, and this contains the aldehyde of vitamin A. Also, when either visual purple or visual yellow is treated with certain concentrated mineral acids, a number of coloured pigments are produced which, like visual purple, are bleached by ordinary white light, and which have narrow absorption bands in a particular part of the visible spectrum.

Cones do not contain visual purple, and their sensitivity to colour is explained by the assumption that the retina contains three different types of cone cells—one type sensitive to red, another to green, and the third to blue-violet light. When these are equally stimulated, white light is produced, and nearly all colours can be produced by mixing

* Vitamin A has a four-unit isoprenoid structure with twenty carbon atoms.

red, green, and blue–violet spectral lights in suitable proportions. These three different types of receiving cells enable the eye to perceive 150 different hues, instead of 150 different types of cells being necessary. This trichromatic (Young–Helmholtz) theory explains many of the facts of colour vision and colour blindness. It is, however, open to serious objections, which were discussed in a Mercer lecture[1], and it is now generally agreed that subjective impressions of colour cannot fully be accounted for without an extension of the trichromatic to a polychromatic theory.

DEFECTIVE COLOUR VISION

With Young's theory as a basis for explaining various types of colour vision, persons of normal vision are known as *trichromats*, i.e. having three types of receptor. Where only one of these receptors is functioning, no variations in hue can be observed, and complete colour blindness results. This is extremely rare.

Dichromats, i.e. those who have two types of receptor, are more common, and they may be divided into three groups—

(*a*) Protanopes or red-blind, i.e. red receptor not functioning

(*b*) Deuteranopes or green-blind, now thought to be due to fusion of red and green receptors

(*c*) Tritanopes or blue-blind.

Tritanopia (*c*) is very uncommon[2], and is of no practical importance. The few cases that have been investigated seem to show that the failure of the blue–violet mechanism is brought about by disease or drugs. A recent investigation[3] suggests that its incidence varies between 1 in 13,000 and 1 in 65,000.

The other two forms—protanopia (*a*) and deuteranopia (*b*)—are, however, quite common, and are estimated to apply to 2–3% of the male population, with the green-blind comprising the larger of the two groups. These types of colour blindness are hereditary, being due like hæmophilia to sex-linked recessive genes, and occurring on the male side of a family but very seldom indeed on the female side. They are transmitted through the female, who acts as a "carrier". Children of the marriage of a carrier woman with a normal man would include a son and a daughter having normal vision along with a carrier daughter and a colour-blind son. From the marriage of a colour-blind man with a woman of normal vision, all sons would have normal vision but all daughters would be carriers. In the unusual case of a carrier woman marrying a colour-blind man, any daughters would be either carriers or colour-blind, but sons might be either normal or colour-blind.

It is, perhaps, unnecessary to add that colour blindness cannot be corrected or cured by wearing spectacles or coloured glasses. In practice there is a marked similarity in the vision of red-blind and green-blind people. Both types view yellows and blues correctly but have difficulty with reds and greens, these two colours being frequently confused with each other. These two types of dichromatic vision are, therefore, frequently referred to as "red–green blindness", and the above estimate of the incidence of colour-blindness refers to this combined (red–green) basis. Actually the two types—protanopes and deuteranopes—do differ somewhat in their colour perception, and various tests have been devised for distinguishing between them. To red-blind people the red portion of the spectrum appears black or dark grey, and there is also a grey band in the bluish-green region separating the yellow from the blue. Green-blind people also see tones of grey in the green region and have difficulty with purple, which also appears grey.

There are various degrees of red and green blindness, and there are the anomalous trichromats[2], who have normal vision except that either the red or the green receptor is lacking somewhat in sensitiveness. This also is more noticeable among males than among females, and may be regarded as a link between normal vision and red–green blindness.

Probably about 8% of males have defective colour vision if the anomalous trichromats are included.

Thus there are different types and degrees of colour-blindness, which makes detection and testing a most difficult matter. This is especially the case with milder forms, such as those shown by the anomalous trichromats, many of whom probably do not realise that they are colour-blind at all. Yet in many respects they are much more dangerous than more extreme cases, as they can see red and green in large and vivid patches. If they are engaged in occupations such as road transport or railway locomotive driving, they can probably see traffic or signal lights in clear weather but fail to distinguish them when the illumination is poor owing to smoke, fog, or mist.

Because of the variation in colour sensitivity between different persons it is impossible to define normal colour vision. It will be seen that it is extremely difficult to draw a clear line between those who possess "normal" colour vision and those whose vision is defective. Even so-called normal observers exhibit slight variations in sensitivity, which are readily detected by means of the Donaldson colorimeter or even by viewing ranges of patterns dyed with specially selected dyes. The term "colour-blindness" is rather a misnomer, and in the light of modern knowledge should certainly be replaced by the title of this paper.

Naturally, whilst extreme cases of defective colour vision are relatively easy to detect, milder forms become progressively more difficult, and several tests may be involved. The difficulty of detection is increased by the deceptions practised by the sufferers and by the ingenuity they often display in concealing their defect. They cannot be blamed for this, as in some cases discovery would probably result in dismissal, or at least in transfer to another job which they might not desire. The present author detected abnormal colour vision in a young man who for some years had been a percher for a large firm of worsted manufacturers. The youth had suspected that there was something wrong, but he knew the types of cloth in the limited range of colours in men's wear, and by listening to

the shade names given by other employees he had succeeded in carrying out his duties to the complete satisfaction of his employers.

A good case can be made out for making the testing of colour vision a routine matter in the medical inspection of older schoolboys or by Youth Employment Officers and others who are advising juveniles regarding careers where accurate judgment of colours is essential [4]. It is surprising to learn that no such provision is made in the National Health Insurance Acts in regard to testing of eyes. The City and Guilds of London Institute in their syllabuses for the Dyeing of Textiles do insert a note for the guidance of teachers recommending that every student should be tested for his colour perception early in his course.

TESTS FOR DEFECTIVE COLOUR VISION

Some tests for abnormal colour vision have been mentioned previously in this *Journal* [5], and need only a brief reference.

The Holmgren Wool Test

This is one of the earliest tests and was reported in this *Journal* many years ago [6]; it is criticised on account of lack of sensitiveness.

The Edridge-Green Lantern Test

The apparatus is a kind of lantern on the front of which are a number of discs which can be rotated. These discs carry differently coloured glasses, and by fixing one disc and placing one or more of the others in front of it, many varied coloured effects can be thrown on to the screen. The colours may represent normal illumination, or by the addition of neutral-tinted glasses they can correspond to signal lights as the latter appear in fog, rain, or mist. This helps to catch out the anomalous trichromats, who have been justly described as "dangerous colour-blinds". This kind of test is used by the Board of Trade, British Railways, and many shipping companies.

The Ishihara Test [7]

This test employs a series of cards on which numbers in different colours are formed on a background of coloured spots. It is widely used for testing colour vision in various branches of the armed forces, it gives reliable results [4], and it is simple and convenient to use. Among the ingenious plates in this series are six (No. 22–27) which distinguish between red-blindness and green-blindness. Each of the four plates 22–25 contains two numbers, one coloured scarlet and the other purple. Red-blinds fail to see the scarlet and read a single (purple) number; green-blinds cannot see the purple, but easily read the remaining (scarlet) number. People having normal vision read the two numbers in each case. Plates 26 and 27 use the same idea with wavy lines instead of numbers.

The Farnsworth Test

This test was described in detail in this *Journal* [1], with explanatory diagrams, but may be summarised as under.

Eighty-five standard colours from the Munsell colour system are copied as pieces of painted card. The testee is expected to arrange the numbered and coloured chips in a circle in the correct sequence. Persons with normal vision show few or no transpositions from the correct order, and the chords joining consecutive numbers lie close to the circumference of the circle. Protanopes (red-blind) give many transpositions; e.g. red (No. 13) is placed next to blue–green (No. 4), and the chords to these numbers go across the circle in a S.E. to N.W. direction. Deuteranopes (green-blind) make similar transpositions, but in this case the chords lie S.W. to N.E. Tritanopes (blue-blind) give rise to chords which run practically horizontal. Thus the three types of colour-blindness can readily be distinguished by using a test which seems to be particularly suited to textile and dyeing requirements.

The Garner Test [5]

Garner has pointed out that the detection of slight abnormalities in colour vision is of very great importance in the dyeing industry, where a difference in passing shades is frequently shown by the foreman dyer compared with, say, his young assistant. Yet both would be regarded as possessing normal colour vision using the tests previously described. The explanation for this has already been given, and the defect is equivalent to a slight blue-blindness in older people, although produced in an entirely different manner from that of tritanopes. Artificial light (from metal-filament electric lamps) is somewhat yellowish, so that people having the above characteristics see colours in daylight as they are seen by more normal-sighted persons in artificial light. Based on these facts, tests for this form of defective colour vision have been devised using patterns which change in hue in artificial light as compared with daylight. Garner's test is one of the best known of these, and it is extremely useful for detecting small deviations from normal colour vision such as are likely to occur between foremen dyers and their assistants or between the various members of the staff of a dyeing laboratory.

The Wilson Test

Considerable scope exists for the development of the idea of using patterns which change in shade in artificial light as a means of comparing the colour vision of dyehouse employees from the standpoint of practical colour matching. The present author is indebted to Mr. E. Wilson, of the Central Laboratory, Bradford Dyers' Association Ltd., for a range of patterns which seem remarkably sensitive for this purpose. They consist of a series of browns dyed on worsted cloth ranging from reddish-brown to greenish-brown, all of which tend to redden in artificial light. A standard brown pattern dyed with dyes which turn green in artificial light is then placed in contact with the range of browns previously numbered, and a match is made in standard "artificial sunlight". The number corresponding to the standard pattern is noted.

The Glenn Colorule [8]

This is a kind of slide rule which embodies the same principle as the previous test. A series (OB)

of twenty-four shades dyed on wool ranging through blue–grey–yellowish brown is mounted alongside another series (VY) ranging through violet–grey–yellowish green. The OB series turns greener and the VY series turns redder in artificial light. For testing observers, the testee, using an unvarying light source, opens up the slide rule and slides one series with respect to the other, until the point is found where adjacent OB and VY samples give the closest possible match. For recording purposes each pattern in the OB series is numbered consecutively 1–24, whereas the VY series is lettered A–X. The instrument can also be used for comparing light sources using the same procedure, except that a single observer views the samples under the various light sources instead of several observers viewing the samples under one light source.

(*MS. received 1st October 1952*)

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[5] Garner, J.S.D.C., **58**, 65 (1942).
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[7] Ishihara, S., *Tests for Colour Blindness* (10th edition 1951. Nippon Isho Shuppan Co. Ltd. London: H. K. Lewis & Co. Ltd.)
[8] The Glenn Colorule may be obtained from the Research Laboratory, Sidney Blumenthal & Co. Inc., Shelton, Conn., U.S.A.

COMMUNICATION

The Dyeing of a Blend of Wool and Fibrolane for the Hand Knitting Trade

R. C. CHEETHAM

The dyeing of a hand knitting yarn consisting of a blend of wool and Fibrolane has been investigated with a view to providing a method of obtaining dyeings of adequate wet fastness and softness of handle.

Preliminary work on separate fibres showed the effect of acid solutions as used in normal wool-dyeing methods on Fibrolane, and indicated the necessity of employing modified dyeing techniques if degradation of the fibre were to be avoided. It was found that pH control, and to a lesser degree temperature control, were essential, and as far as possible dyeing should be conducted at pH 4·0, using buffered solutions for the purpose; under these conditions imbibition by Fibrolane of water from acid solutions is at a minimum.

Rate-of-dyeing curves for Fibrolane, wool, and a blend of the two, at various pH values between 2·0 and 7·0, show that Fibrolane has a high initial rate of absorption of dye at the lower pH values in this range; in a dyebath at pH 4·0 the absorption of acid is such that the final pH approaches neutral where acid is used alone, but with buffered acid solutions the pH can be stabilised much more readily, and remains about pH 4·0.

Dyeing at pH 4·0 is most effectively conducted with a buffered solution based on an acid with a pK_a value around 4, e.g. lactic, formic, or acetic acid.

It was found necessary to use aggregated acid dyes to obtain the requisite wet fastness, which remained satisfactory with the modified method. Suggestions are made for assisting level dyeing.

Introduction

One of the uses for Fibrolane (a regenerated protein fibre) lies in hand knitting yarns, where a desirable feature is the softness of handle normally obtained only with the finer qualities of wool [1]. Casein fibres may be blended with wool, e.g. 56s quality, usually in the proportion of 30% to 70% wool, to give a yarn which retains the appearance and handle of a fine-quality yarn. In dyeing such a blend, a primary requirement is to preserve the features contributed by Fibrolane.

The effects of scouring and dyeing involve changes in physical properties of the fibre, including increase in water imbibition; this is particularly noticeable with Fibrolane, and in certain cases degradation of the fibre structure may take place owing to severity of the treatment.

Change in physical condition of regenerated proteins during dyeing is mainly a function of their constitution, which differs from wool in the absence of the cystine linkages which contribute to the chemical resistance of wool. Attempts have been made to remedy this by modifications in the manufacture of Fibrolane [2]. The fibre now in commercial production incorporates such modifications, which have been found to increase the resistance to hot dyebaths. It is still necessary, however, to adopt modified techniques for dyeing so as to preserve as far as possible the optimum condition of the fibre during dyeing.

The dyeing experiments described in this paper refer to the application of acid dyes, but it is intended to extend the investigation to other classes of dye. For hand knitting yarns made from wool the levelling acid dyes have been widely used, and whilst in light shades the resultant fastness may be adequate, it has been found that this class of dye is not sufficiently fast to washing in many shades on Fibrolane, particularly in view of the modern tendency to insist on a higher standard of performance in the final product. It is known that casein fibres dyed with levelling acid dyes have in general inferior fastness to wool [3]. The few exceptions are those dyes possessing good neutral-dyeing properties, but these are usually deficient in light fastness and do not in any case provide a satisfactory range for normal requirements. It is therefore necessary to employ the aggregated type of acid dyes on blends of wool and Fibrolane. The application of these dyes has been studied from the following aspects—

(1) Handle of the dyed yarn
(2) Solidity of shade on wool and Fibrolane
(3) Adequate wet fastness on Fibrolane

(4) Level-dyeing properties of the dyes

In the first place it seemed clear that dyeing problems concerning Fibrolane blends could be elucidated only by examining Fibrolane itself prior to blending, for it was believed that the ultimate performance of the blend was controlled largely by the behaviour of the constituent fibres.

In studying the properties of Fibrolane, the first concern was to investigate the action of dye liquors such as are met in normal wool dyeing methods for acid dyes; under such conditions it was assumed, and later confirmed, that the wool fibre would not be materially affected.

Experimental

PRELIMINARY WORK ON HANDLE AND IMBIBITION

Preliminary work on the separate fibres prior to blending established a connection in the case of Fibrolane between loss of handle and change in physical properties during treatment with acid solutions such as are met in normal wool processing. It was found that pH of the solution was one of the most critical factors. The nature of the acid and the temperature of treatment were also found to have some influence on condition of the fibre.

For these preliminary experiments a range of acid solutions was prepared covering pH values one would expect to find in acid dyebaths for wool. In order to obtain thorough penetration of the fibres, continuous tow was used in place of yarn, and for each test 5 g. of 3¼-denier Fibrolane was immersed in 100 c.c. of various solutions at pH values varying from 2·0 to 7·0 for 1 hr. at 80°C., 95°C., and the boil. After this they were rinsed, dried at room temperature, and examined as individual lots in order to assess the effect of pH on physical condition, particularly as regards hardening of the fibres.

In the first place the examination was qualitative, relying on an assessment of the handle of the fibres in each lot. It was noted, and confirmed by different assessors, that appreciable hardening of the fibres was produced in solutions at the lower pH values in the range used, viz. 2·0–3·0, and to a lesser extent at pH 5·0–7·0 at the other end of the range, the fibres retaining their condition best at pH about 3·5–5·0. Hardening was greatest at the boil, and was less noticeable as the temperature was lowered.

In order to measure the hardening effect more precisely, it was decided to examine water imbibition, since this has been found to be one of the physical phenomena closely associated with dyeing. The experiments described above were repeated in tubes using a thermostatic oilbath, and constant volume was maintained during the time of immersion. Water imbibition was measured by taking samples of the treated tow of Fibrolane and centrifuging for 5 min. at 3000 r.p.m., i.e. acceleration towards centre = 1200 ***g***, before weighing and drying at 55°C. to constant weight. When imbibition was plotted against final pH, it was found that the curves were parabolic, with minimum imbibition corresponding to a final pH of approx. 4·0. High imbibition resulted when fibres were immersed in solutions giving final pH values at both ends of the range 2·0–7·0.

As relatively high imbibition implies a definite tendency to fibre degradation due to swelling, it is clearly advantageous to conduct dyeing at pH 4·0. In order to maintain the pH at such a precise value, it seemed essential to use buffered solutions to allow for the possible variations in pH one might expect under practical dyebath conditions. Later experiments showed that, as the temperature of treatment in acid solution was lowered, so the effect on the fibre was correspondingly reduced, and treatment in the cold increased the permissible pH range to 2·0–5·0.

Parallel determinations of water imbibition of 64s wool sliver indicated that pH and temperature had a much less critical effect on the water imbibition of wool over the same pH range.

RATE OF DYEING OF AN AGGREGATED ACID DYE

It seemed desirable to investigate these factors in relation to rate of dyeing. A representative aggregated acid dye, viz. Alizarine Brilliant Green G conc. (LBH), was selected for a series of dyeing tests using various acids at three different pH values, on the separate fibres and also on a blend. The pH values selected were not entirely arbitrary but were based on the pH range used in the preliminary experiments: they included the pH found to be most beneficial for fibre condition and that at each end of the range, viz. corresponding approx. to 2·5, 4·0, and 6·0. The dyeings, 1% in each case, were conducted in tubes in a thermostatic oilbath, and the dye liquor was maintained at constant volume. The patterns were agitated periodically. Dyebath exhaustion was calculated from Spekker absorptiometric readings on samples of dye liquor removed at 5-min. intervals.

Dyebath conditions were standardised by entering the material at the same initial temperature of 35°C. for each test, the dyebath being raised gradually to 95°C. in each case. This procedure accords with normal practice for the application of aggregated acid dyes to wool.

The materials used in all the tests were 2/24s worsted yarn, 2/18s 4½-denier 6-in. staple Fibrolane, and a 3/15s blend yarn composed of 70% wool and 30% Fibrolane. For each test 2·5 g. of yarn was used at a liquor ratio of 50 : 1. Before being dyed the three yarns were scoured together with a non-ionic synthetic detergent and rinsed before being entered into the dyebath.

RATE OF DYEING IN DYEBATHS USING NORMAL METHODS

The first step was to investigate normal methods of dyeing, so dyebaths were prepared using formic acid at pH 2·6, acetic acid at pH 3·7, and ammonium acetate at pH 6·7. Fig. 1 shows dyebath exhaustion at pH 2·6 plotted against time, and indicates the degree of exhaustion during the first hour in each case; Table I gives the change in pH after dyeing has proceeded for the same length of time.

At low pH Fibrolane has a high initial dye absorption and a fairly high rate of dyeing, the dyebath being almost completely exhausted after

30 min. Wool has a much lower initial absorption and a lower rate of dyeing during the first 30 min., the rate being similar to Fibrolane during the next 20 min. as the temperature rises, and reaches the same degree of exhaustion in 50 min. as Fibrolane does in 30 min. The blend follows a similar course to Fibrolane, but with a somewhat lower initial absorption, the dyebath arriving at a similar state of exhaustion in only slightly longer time than for Fibrolane alone. There is no change in dyebath pH during dyeing in the case of wool, a noticeable rise with Fibrolane, and a slight rise with the blend. These results indicate that at low pH Fibrolane blended with wool in the proportion of 30 : 70 influences dyebath pH only slightly but exhaustion markedly. This illustrates the high absorptive capacity for dye of Fibrolane in dyebaths at low pH.

TABLE I

Change in pH of Dyebaths based on Various Acids

Acid Solution	Initial pH	Final pH		
		Fibrolane	Wool	Blend
Formic acid	2·6	2·9	2·6	2·7
Acetic acid	3·8	5·55	4·3	4·9
Ammonium acetate ...	6·7	7·2	6·7	7·0
Phosphoric–phosphate ...	2·55	2·65	2·5	2·55
Phosphoric–phosphate ...	3·85	7·1	6·4	6·6
Phosphoric–phosphate ...	5·97	7·2	6·8	6·9
Phthalic–phthalate ...	2·5	2·57	2·5	2·5
Phthalic–phthalate ...	3·9	4·3	4·1	4·15
Phthalic–phthalate ...	6·05	6·1	6·11	6·12
Lactic–lactate	3·87	4·15	4·0	4·05
Acetic–acetate	3·8	3·85	3·8	3·83

Fig. 2 shows the effect of dyeing at pH 3·7, and again Fibrolane has a higher initial rate of absorption than wool. Dyeing proceeds steadily for 50 min., after which the rate of dyeing slows down; exhaustion is not complete after a dyeing time of 1 hr., being much less than at pH 2·6. Wool shows a similar result to Fig. 1. The blend follows the course shown by wool in the earlier stages of dyeing, but later there is a tendency for dyeing to slow down and the course of the graph corresponds more closely to that of Fibrolane. The final pH of the dyebath shows a striking increase for Fibrolane, but in the case of the blend the pH rises much less.

At pH 6·7 (Fig. 3) the rate of dyeing in all cases is low, with exhaustion no more than 30%. It will be noted that the rate of dyeing of Fibrolane at this pH is consistently low. The final pH of the dyebath is around the neutral point in all cases.

This first series of graphs indicates that at low pH a Fibrolane–wool blend, containing 30% of the former, dyes similarly to Fibrolane; but dyebath pH is not unduly influenced during a dyeing time of 1 hr., since the amount of acid extracted is insufficient to cause a significant rise in pH.

At pH values of 3·8 and over the blend progressively approaches wool in behaviour, since Fibrolane influences dyebath pH to a much greater extent and the dye-absorptive capacity of Fibrolane is lower than that of wool as the pH rises.

RATE OF DYEING IN BUFFERED SOLUTIONS

Various buffered solutions, based on standard laboratory mixtures[4–6], were prepared, and the pH adjusted to conform approximately with the three values used in the first series of experiments. Fig. 4–6 illustrate the results obtained in phosphoric acid–phosphate buffers at pH 2·55, 3·85, and 5·97.

At the lowest pH the results are very similar to those with formic acid at about the same pH. The graphs for higher pH show low exhaustion rates, and it will be noted that the final dyebath pH is not far from 7·0. The absorption of acid, resulting in a high dyebath pH, is not in this case confined to Fibrolane but is shared also by wool, presumably owing to the relatively high affinity of the phosphate ion for protein.

Phthalic acid–phthalate buffer solutions at pH 2·5, 3·9, and 6·05 indicate that rates of dyeing at pH 2·5 and 3·9 are both similar in form to results given by phosphate buffers at pH 2·55. This buffer appears to give better control over dyebath pH (Table I) than does the phosphoric acid–phosphate buffer, possibly on account of the difference in ion affinity between phthalic and phosphoric acids.

Buffer solutions based on lactic acid and acetic acid respectively are considered next (Fig. 7 and 8), and one of the differences between them is found in a rather more gradual rise in dyeing rate in the case of lactic acid, so that maximum exhaustion is reached in 40 min. as against 30 min. for acetic acid. The rise in dyebath pH is slightly more pronounced with the former acid.

These experiments show that dyebath pH can be satisfactorily maintained constant by the use of various kinds of buffered solutions, but that the efficacy of a particular buffered acid depends partly on its affinity for protein.

As regards the actual preparation of the buffered solutions, it was found possible to amend the standard mixtures, and the amounts of the constituents were reduced so as to provide only sufficient undissociated acid in the system to allow for absorption by the fibre and still retain the advantages of buffered solutions. For example, the acetic acid–acetate buffer could be made up with a quarter the amount of acid recommended for a standard buffer solution without affecting the final pH to any great extent after immersion of the fibre. The lactic acid–lactate buffer was adjusted similarly, and the precise quantities of constituents used for two of the buffer solutions were as follows—

ACETIC ACID–ACETATE BUFFER (pH 4)

198·2 c.c.	0·2 N. Acetic acid per litre
35·7 c.c.	0·2 N. Sodium hydroxide per litre

LACTIC ACID–LACTATE BUFFER (pH 4)

1·0 c.c.	Lactic acid (sp. gr. 1·21) per litre
0·625 g.	Sodium bicarbonate per litre

EFFECT OF DYEBATH pH ON HANDLE OF THE YARN

It was found that the handle of the Fibrolane yarn, and also that of the blend, deteriorated when the dyebath pH was allowed to rise above 5·0, and the pH was influenced in turn by the absorption of acid by Fibrolane. It has been shown that Fibrolane has minimum swelling and hence lowest water imbibition at or around pH 4·0, and there

exists a range of pH which is critical as regards the physical condition of the fibres. Consequently it is possible to correlate dyebath pH with physical condition of the fibre.

DYE AFFINITY AND DISSOCIATION CONSTANTS OF ACIDS

In view of the results obtained with various acids and the observed differences in physical condition of the dyed fibres, it seemed possible that absorption of acid by Fibrolane would be related to degree of dissociation of the acid. Accordingly a number of acids were arranged in descending order of their dissociation constants[7], as shown in Table II.

TABLE II

Dissociation Constants of Acids

Acid	Dissociation Constant K_a	$pK_a = -\log K_a$
Orthophosphoric* ...	$1{\cdot}1 \times 10^{-2}$	2·0
Phthalic*	$1{\cdot}26 \times 10^{-3}$	2·9
Citric*	$8{\cdot}4 \times 10^{-4}$	3·1
Formic	$1{\cdot}76 \times 10^{-4}$	3·7
Lactic	$1{\cdot}38 \times 10^{-4}$	3·9
Succinic*	$6{\cdot}6 \times 10^{-5}$	4·18
Benzoic	$6{\cdot}3 \times 10^{-5}$	4·2
Acetic	$1{\cdot}75 \times 10^{-5}$	4·7
Butyric	$1{\cdot}48 \times 10^{-5}$	4·8

*First dissociation constant

Since minimum imbibition coincides with pH 4·0, and as in practice the physical condition of Fibrolane, as of any other fibre, is closely connected with fibre swelling, it was concluded that optimum results after applying a particular process could be expected by maintaining the pH of the liquor at or near this value. This can clearly be more conveniently done by using buffered solutions, and, further, if an acid be used having pK_a about 4, then such an acid will provide the most effective way of maintaining dyebath pH at the required value.

For practical reasons, only acids available commercially were considered, and these included formic, lactic, and acetic acids, of which lactic acid with a pK_a value close to 4 appeared to give the most satisfactory results. Acetic acid was, however, also suitable from the point of view of retaining the handle of a blend yarn composed of Fibrolane and wool.

Practical Dyeing Considerations

Having indicated the dyeing method, it seemed necessary to observe the behaviour of aggregated acid dyes in the practical dyeing of blend yarns. The chief desiderata are solidity of shade on the two fibres, fastness to washing of the dyed yarn, and level dyeing properties.

Fig. 1–10

Exhaustion Curves of 1% Alizarine Brilliant Green G conc. from Various Dyebaths

△ Fibrolane BX □ Wool ○ 70% Wool + 30% Fibrolane BX

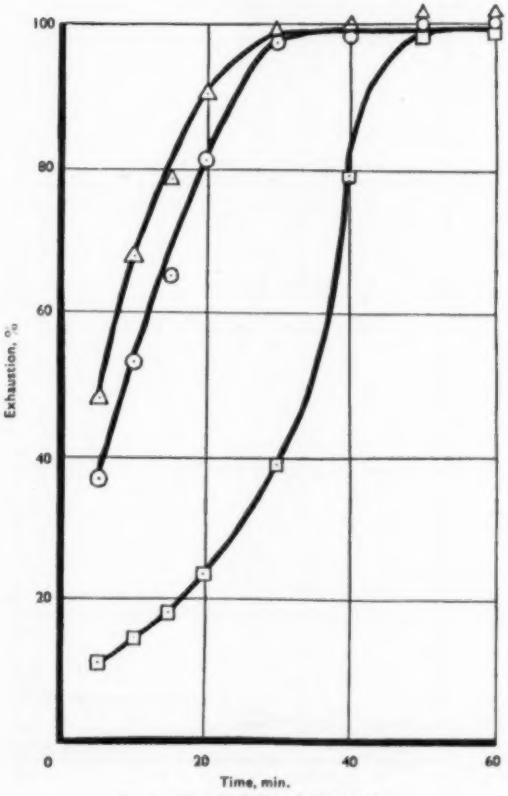

FIG. 1— Formic Acid at Initial pH 2·6

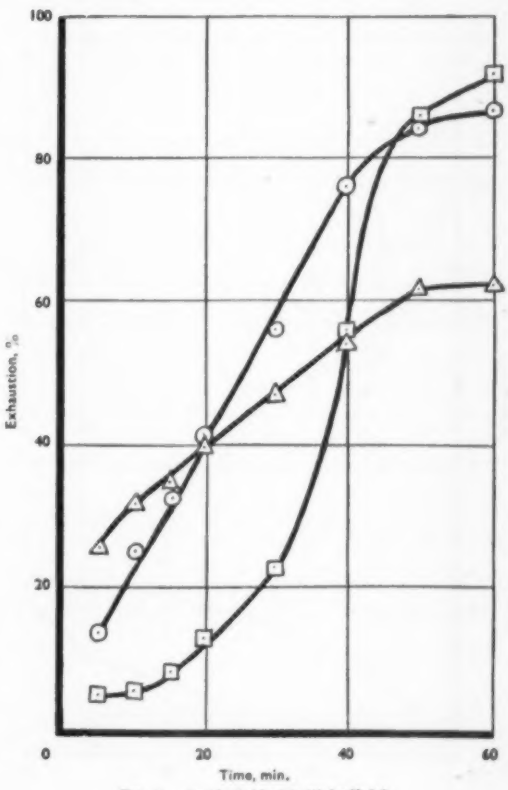

FIG. 2 — Acetic Acid at Initial pH 3·7

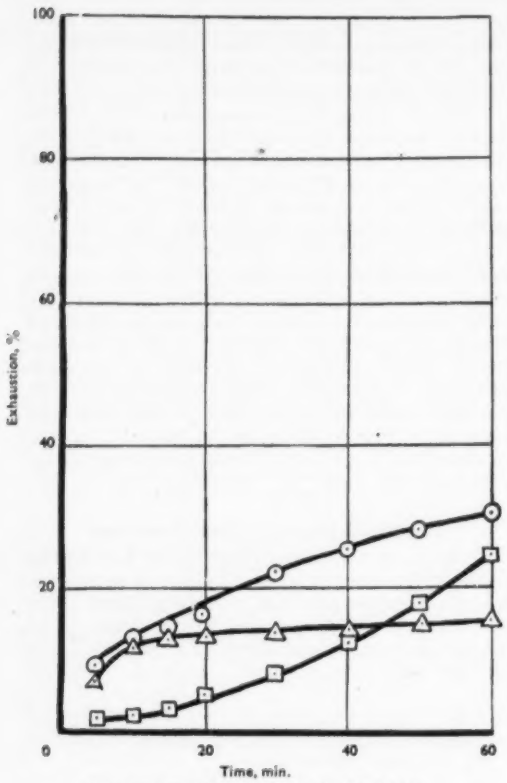

FIG. 3— Ammonium Acetate at Initial pH 6·7

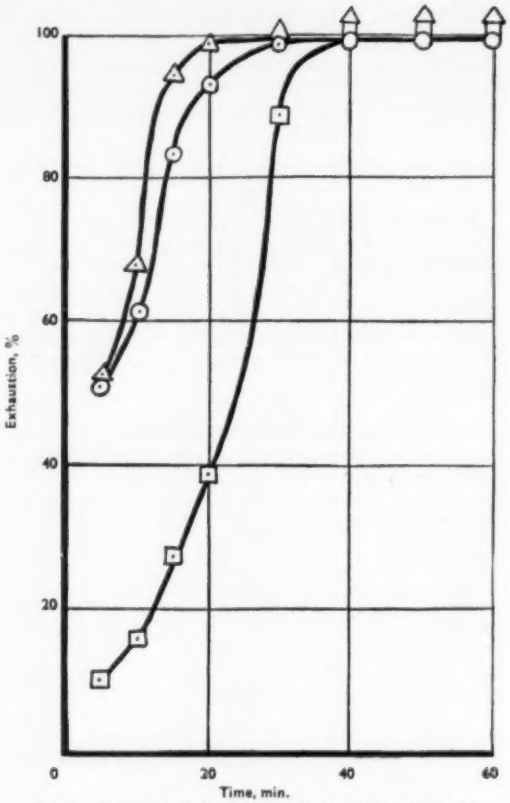

FIG. 4— Phosphoric Acid–Phosphate Buffer at Initial pH 2·55

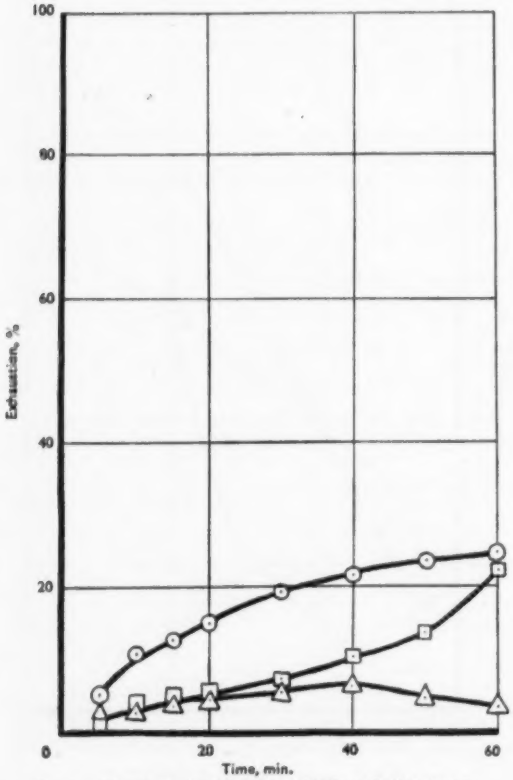

FIG. 5— Phosphoric Acid–Phosphate Buffer at Initial pH 3·85

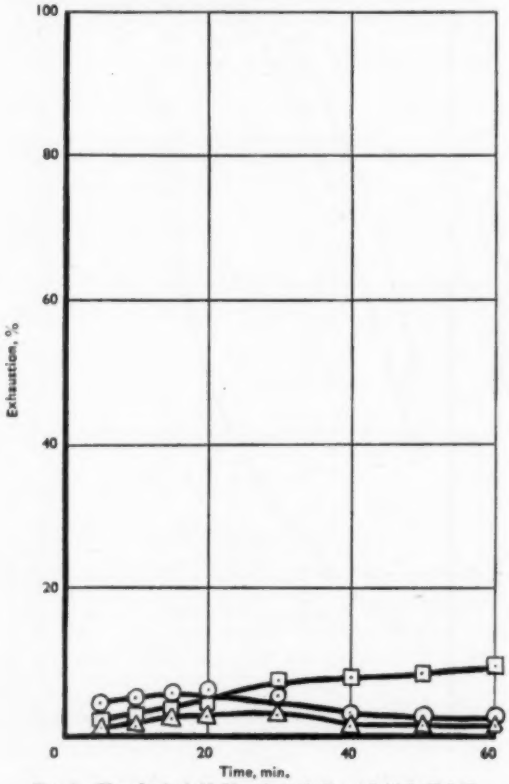

FIG. 6— Phosphoric Acid–Phosphate Buffer at Initial pH 5·97

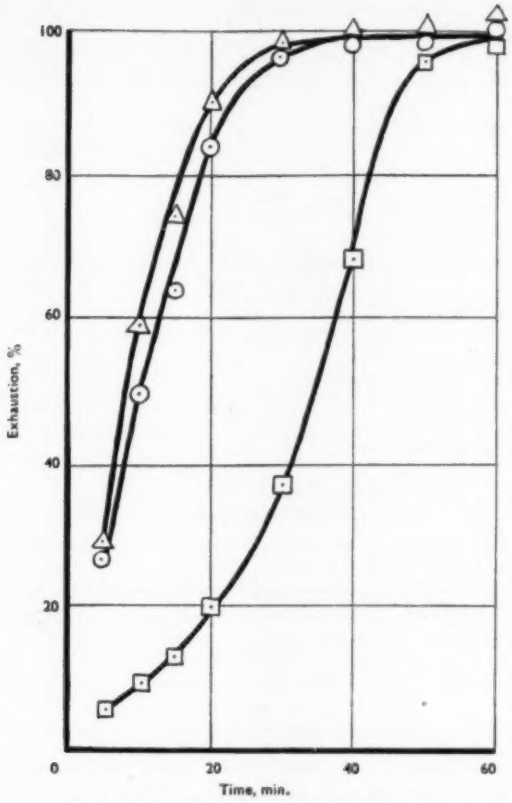

FIG. 7— Lactic Acid–Lactate Buffer at Initial pH 3·87

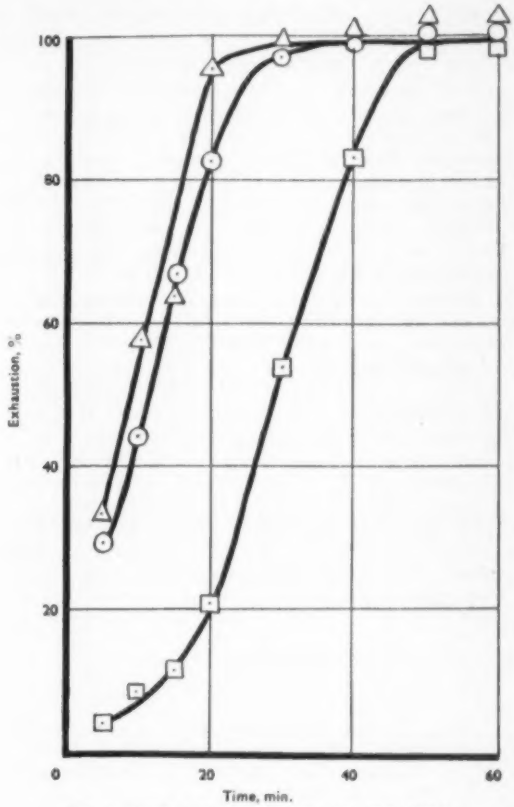

FIG. 8— Acetic Acid–Acetate Buffer at Initial pH 3·8

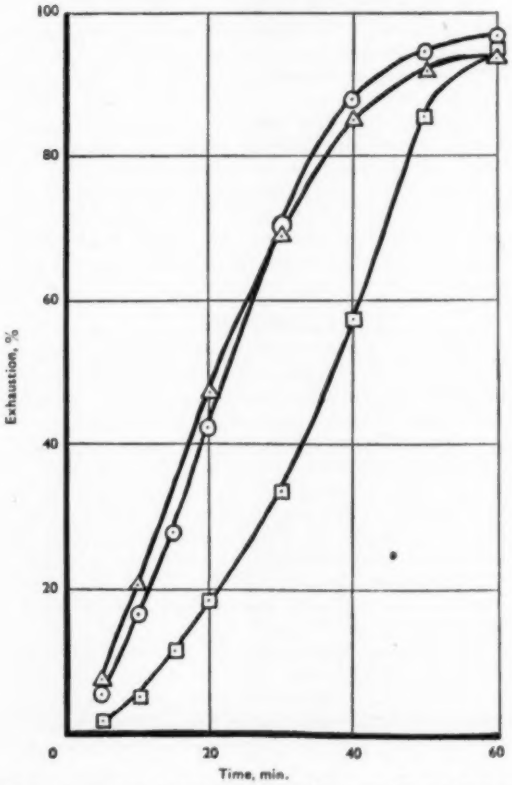

FIG. 9— Acetic Acid–Acetate Buffer + Calsolene Oil HS (3 g./litre) at Initial pH 3·8

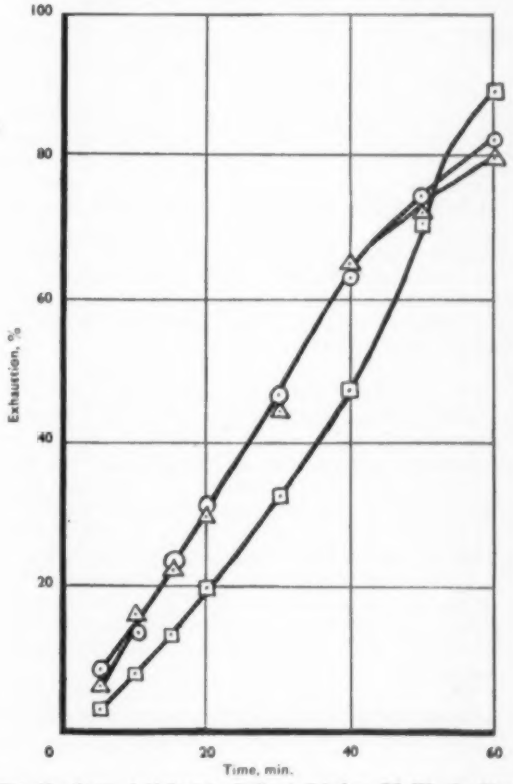

FIG. 10— Lactic Acid–Lactate Buffer + Calsolene Oil HS (3 g./litre) at Initial pH 3·9

SOLIDITY OF SHADE

It is well known that difficulties attend the dyeing of two or more fibres of different dyeing properties where a solid shade is required by a one-bath process. The difficulties are often accentuated where the fibres, as in the case of Fibrolane and wool, have similar dye affinities and only one class of dye is being used. The dyer has to rely on his ability to control the relative rates of absorption on the two fibres.

It has been shown in the rate-of-dyeing experiments that Fibrolane has a higher initial rate of absorption than wool, and maximum absorption is usually attained at a lower temperature. In practice it is sometimes found that dye migrates from Fibrolane to wool in course of time. However, at the pH indicated by the modified dyeing technique or at lower temperatures than the boil, this tendency is not so marked, and to a large extent reliance has to be placed on selection of dyes with least preferential affinity for Fibrolane.

The dyes listed in Table III have been found to offer a basis for producing the best solidity within reasonable dyeing time.

TABLE III

Suitable Dyes for Blends

Carbolan Yellow 4GS (ICI)
Coomassie Yellow RS (ICI)
Coomassie Fast Orange GS (ICI)
Polar Red RS conc. (Gy)
Sulfonine Red G (S)
Carbolan Blue BS (ICI)
Carbolan Crimson 3BS (ICI)
Alizarine Brilliant Green G conc. (LBH)
Alizarine Fast Grey G (CAC)

FASTNESS TO WASHING OF THE DYED BLEND

All the dyes quoted in Table III were found to have adequate fastness for hand knitting yarns. The standard achieved in pale shades was equivalent to S.D.C. No. 2 washing test for wool materials [8], and under the conditions of this test little change of shade was recorded with little or no staining of white materials. In some dark shades, particularly maroons, it may be difficult to maintain the highest standards of fastness, but by using some of the dyes quoted above the fastness of Fibrolane–wool blends compares favourably with that of the same dyes on wool.

LEVEL-DYEING PROPERTIES

The levelling power of aggregated acid dyes in dyebaths at pH 4 would normally give rise to difficulties, particularly in certain types of dyeing machines such as Hussongs. It is observable that the use of a buffered solution reduces the tendency to unlevel dyeing, and levelness is still further improved if a suitable auxiliary agent is added.

The use of auxiliary agents as aids to levelling under the conditions described has been investigated, and some success has been achieved with Lissapol N (ICI) and Calsolene Oil HS (ICI) using 3–5 g./litre.

The effect of Calsolene Oil HS on rate of dyeing is shown in Fig. 9 and 10. It can be observed that a bath based on lactic acid containing 3 g. Calsolene Oil HS per litre appears to provide a method of applying the aggregated type of acid dye to a blend of Fibrolane and wool.

It is probable that other aggregated dyes, in view of the difference in dyeing rates, would require a rather greater concentration of auxiliary agent.

In order to give further aid to the levelling of these dyes, if the yarn is scoured in a non-ionic detergent as suggested above, the scoured yarn may with advantage be entered into the dyebath without rinsing.

It should be remembered that the behaviour of aggregated acid dyes is marked by variations between individual dyes, and this is equally true when the modified dyeing methods are being used. Careful selection of dyes is therefore of paramount importance.

Application of Modified Technique on Semi-bulk Scale

In order to test the efficiency of the modified technique for Fibrolane–wool blends, 5 lb. of 3/15s Fibrolane–wool blend yarn was scoured and dyed in 170 litres, using a Longclose laboratory Hussong machine. Scouring was conducted in a 0·2% solution of Lissapol N at 50°c.

The dye recipe used was as follows—

3% Polar Red RS conc. (Gy)
0·5% Carbolan Blue BS (ICI)

The dyebath consisted of the lactic acid–lactate buffer solution with the addition of 5 g. Calsolene Oil HS per litre.

The yarn, after scouring, was entered at 35°c. and raised to 95°c. during one hour, dyeing being continued at this temperature for a further hour.

The dyebath exhaustion obtained after this period was 92·9%.

Assessment of the results showed satisfactory handle, good solidity of shade on wool and Fibrolane, a reduction in tensile strength due to dyeing of 7·5%, and a shrinkage in skein length of 10·42% (measured without tension). From a commercial point of view this small trial could therefore be considered satisfactory for a wool blend.

Discussion

Fibrolane possesses intrinsic properties which characterise its dyeing behaviour, so that if the desirable qualities such as softness of handle are to be retained in blends, modification of normal wool-dyeing techniques will in many cases be necessary.

Physical properties relevant to the dyeing process have been measured, and attempts made to correlate water imbibition and dyebath pH with the handle of Fibrolane over a range of pH values. It was found that minimum imbibition and optimum physical condition of the fibre corresponded to pH 4·0 when processed at temperatures between 90°c. and the boil.

In studying the application of acid dyes, it should be remembered that one of the distinctive properties of Fibrolane is the ability to absorb acids at low temperature, and this in turn influences dyebath pH. For blends with wool the extent to

which the final pH of the dyebath is influenced depends on the initial pH, and to some extent on the amount of Fibrolane present in the blend.

At an initial pH of 2·0 the amount of acid absorbed by the fibre does not materially affect the final pH of the bath, since the amount so absorbed is a relatively small proportion of the total available acid. The pH thus remains appreciably lower than 4·0.

Dyebaths set at pH 4·0, however, using acid only, become depleted of acid, and the pH rises above 4·0.

In order to maintain dyebath pH within the desired range it is necessary to use buffered solutions, and although in general the tendency for pH to rise is thus reduced, it is by selecting a buffered solution based on an acid with a pK_a value as near to 4 as is practicable that the possibility of dyebath pH shifting outside the critical range is almost entirely removed.

The practical application of these principles to the dyeing of a blend of wool and Fibrolane knitting yarn has been demonstrated on a laboratory scale for a range of shades and in a small Hussong dyeing machine for a maroon shade. Using aggregated acid dyes, a high standard of wet fastness coupled with good solidity of shade on wool and Fibrolane has been achieved.

Work is continuing on a detailed examination of aggregated acid dyes when applied by the modified methods described, and it is intended to arrange bulk trials in due course.

* * *

The author wishes to thank the Directors of Courtaulds Ltd. for permission to publish this paper, Mr. J. Boulton for advice during its preparation, and Messrs. J. Sadowski and R. W. Henley for carrying out most of the practical work.

COURTAULDS LTD.
DROYLSDEN
MANCHESTER

(Received 3rd May 1952)

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[1] Tattersfield, C. P., *Dyer*, **95**, 267 (15th March 1946).
[2] Diamond, C., and Wormell, R. L., *J. Textile Inst.*, **30**, P 224 (1939).
[3] Bird, C. L., *The Theory and Practice of Wool Dyeing* (2nd edition 1950. Bradford: Society of Dyers and Colourists), p. 142.
[4] Clark, W. M., *The Determination of Hydrogen Ions* (2nd edition 1923. Baltimore, Md.: Williams & Wilkins).
[5] Kolthoff, I. M., *pH and Electro-titrations* (New York: John Wiley & Sons Inc. 1931).
[6] Palmer, W. G., *Experimental Physical Chemistry* (Cambridge University Press 1941).
[7] *Handbook of Chemistry and Physics* (32nd edition 1950–51. Cleveland, Ohio: Chemical Rubber Publishing Co.), p. 1507.
[8] *Second Report of the Fastness Tests Committee* (2nd edition 1950. Bradford: Society of Dyers and Colourists), p. 10.

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Adsorption of Metal Ions by Naturally Pigmented Keratin Fibres

In the course of a study of the effect of various metal ion catalysts on the bleaching of pigmented keratin fibres with hydrogen peroxide, an interesting fact has emerged, for measurements of the amounts of iron and copper adsorbed by pigmented and non-pigmented fibres have revealed striking differences. A known weight of purified fibre was immersed at 25·0 ± 0·2° C. in 0·001 M. ferrous sulphate or cupric sulphate solution adjusted to pH 2·41 by addition of sulphuric acid, the solution : fibre ratio being 50 : 1. The amount of iron remaining in the solution after 144 hr. was estimated by the colorimetric method depending upon the formation of a blue–red colour by the interaction of ferrous salts and ammonium thioglycollate, and measurement of the optical density of the coloured solutions at 530 mμ. using a Unicam spectrophotometer. The amount of copper remaining in solution after 144 hr. also was estimated colorimetrically, by means of the yellow complex formed between cupric salts and an ammoniacal solution of sodium diethyldithiocarbamate, measurements of optical density being made at 445 mμ. The results obtained on various pigmented and white keratin fibres are shown in Table I, from which it is evident that the amounts of iron and copper adsorbed by the pigmented fibres are much greater than those taken up by the white fibres. The differences cannot be accounted for in terms of variations in diameter, but appear to be associated with the presence of pigment.

Further, there is an approximately linear relation (Fig. 1) between the degree of coloration of the fibre and the amount of metal adsorbed. It

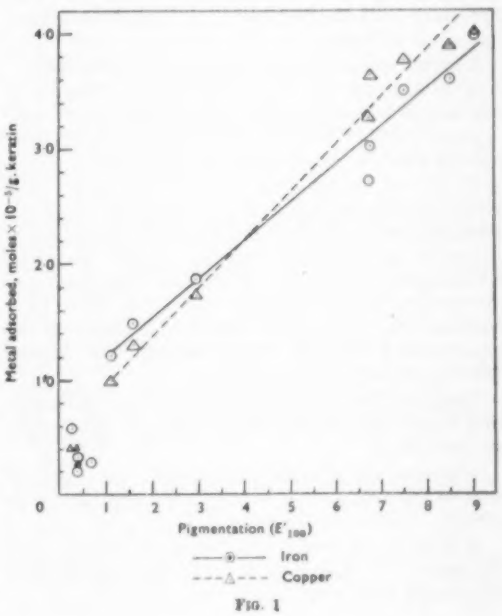


FIG. 1

is difficult to express the colour of pigmented fibres quantitatively, but in this investigation the following technique was adopted. 0·2 g. of the dry fibre was hydrolysed by refluxing in 75 ml. of 2 N. caustic soda for 8 hr., and the spectral absorption curve of this digest of amino acids and pigment was measured, after dilution to 200 ml. Optical density measurements were made at 25-mμ. intervals between 350 and 1000 mμ. using the Unicam spectrophotometer. All the curves obtained by plotting the extinction coefficient (E'_{100}) for 1 g. of keratin dissolved in 100 ml. of 0·75 N. caustic soda against wavelength had the same general shape, and it was found convenient to use the value of E'_{100} at 450 mμ. as an index of the colour of the solution. It is interesting to note that a similar correlation was found to exist when the "colour" of the solutions was expressed in terms derived from the ultraviolet absorption spectra between 275 and 295 mμ.

TABLE I

Type of Fibre	Colour E'_{100}	Metal adsorbed (moles $\times 10^{-6}$ per g. keratin) Iron	Copper
Black Welsh (No. 1) ...	9·019	3·98	4·01
Black alpaca	8·547	3·60	3·89
Black Welsh (No. 2) ...	7·534	3·50	3·76
Yorkshire-grown Leicester...	6·797	3·02	3·63
Black goat hair	6·754	2·71	3·26
Dark brown human hair ...	2·966	1·87	1·72
Medium brown human hair	1·596	1·48	1·30
Light brown human hair ...	1·112	1·21	0·99
White Welsh wool ...	0·391	0·34	0·28
Merino wool	0·367	0·20	0·40
White alpaca	0·250	0·58	0·40
White Yorkshire-grown Leicester	—	0·43	0·22
White goat hair	—	0·13	0·21
White human hair	0·674	0·28	—
White Lincoln wool ...	—	0·22	0·20

The mechanism of the association between the pigmented fibre and the metal ion has not yet been elucidated, but it is tempting to suggest that the pigment itself is concerned. The union is reasonably firm, for if a pigmented wool which has been immersed in ferrous sulphate solution of pH 3 for 45 hr. is washed for 36 hr. in a dilute acid solution of pH 2·4, it still contains $2{\cdot}82 \times 10^{-5}$ mol. of iron per gram of wool, a value which is only very slightly different from that obtained by analysing the ferrous sulphate solution at the beginning and at the end of the adsorption period.

The authors are indebted to the International Wool Secretariat for financial aid which enabled this investigation to be carried out.

G. LAXER
C. S. WHEWELL

DEPARTMENT OF TEXTILE INDUSTRIES
THE UNIVERSITY
LEEDS 2

6th January 1953

Notes

Proceedings of the Council

At a meeting of the Council held at the Offices of the Society, 19 Piccadilly, Bradford, on 11th February 1953 the proceedings included the following items of interest—

LETTER FROM MESSRS. CHORLEY & PICKERSGILL LTD.— A letter was read from Messrs. Chorley & Pickersgill Ltd. expressing appreciation of the note appearing in the January 1953 issue (p. 24) on their having printed the *Journal* for 50 years.

S.D.C. OF AUSTRALIA— It was agreed to send messages of greeting via Mr. G. S. J. White to the President of the Society of Dyers and Colourists of Australia and to the Chairman of the Victorian Section of that Society. Mr. White (Vice-chairman of our Manchester Section) was due to leave on a business trip to Australia and New Zealand, in the course of which he would attend the Annual Dinner of the Victorian Section.

ANNUAL REPORT— A draft of the Annual Report of Council for 1952 was approved for submission to the A.G.M.

REPORTS ON EXTERNAL BODIES— A précis of reports for 1952 by the Society's representatives on external bodies was adopted.

BALANCE SHEET— The Balance Sheet and Statement of Accounts for the year ending 31st December 1952 were approved.

APPOINTMENT OF SCRUTINEERS— It was resolved to invite Mr. E. Stead and Mr. R. Grice to act as scrutineers should a ballot for the election of a Vice-president and Ordinary Members of Council prove necessary.

MR. W. KERSHAW— The resignation of Mr. W. Kershaw from the Terms and Definitions Committee was accepted with regret.

MEMBERSHIP— Twenty-three applications for ordinary membership and eight for junior membership were approved.

Meetings of Council and Committees
February

Council— 11th
Publications— 17th
Colour Index Editorial Panel — 26th
Diploma— 20th

Annual Dinner

Lord Calverley will reply to the toast of the guests, to be proposed by Mr. G. F. Wood (Chairman of the London Section).

Deaths

We regret to report the loss by death of Mr. C. W. Bancroft and Mr. L. A. Lantz.

The Second Edition of the *Colour Index*

Orders are invited before 30th June 1953 at special prepublication rates. A descriptive leaflet and an order form containing details of the prepayments scheme were enclosed with the February issue of the *Journal*, and additional copies may be obtained on application to The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire, England. Residents of the U.S.A. and non-British territories of Central and South America should apply to the American Association of Textile Chemists and Colorists, Lowell, Mass., U.S.A.

1952 Index

The Index of volume **68** (1952) of the *Journal* was despatched with the February 1953 issue to members and subscribers who received the *Journal* during 1952. Those who may not have received a copy may obtain one on application to The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire.

Intermediates direct from Coal

Aniline, phenol, toluidine, xylidine, quinoline, and indole have been produced directly since May 1952 at Institute (West Virginia) by the hydrogenation of coal (300 tons per day)[1]. Amounts are much larger than in normal coal or coke-oven tars.

[1] *Chem. Trade J.*, **131**, 1400 (12th Dec. 1952).

Intermediates in Holland

At the Lutterade coke ovens of the Staatsmijnen in Limburg a 600-ton phenol plant is under construction, and a 2000-ton phthalic anhydride plant has recently begun operation. The phenol will be used in part as raw material for caprolactam, to be used for polyamide fibres and moulding materials.

New Books and Publications

Review of Textile Progress

Volume III 1951

Published jointly by the Textile Institute and the Society of Dyers and Colourists. Pp. 590. Price, 35s. 0d. (28s. 0d. to members of the Society or of the Textile Institute).

The third volume of this annual review is about 100 pages longer than the previous one, mainly owing to the inclusion of the following additional chapters—*Synthetic Fibres—Physics and Chemistry* by G. Loasby and A. R. Munden, *Wool Production* by A. B. Wildman, *Sizing and Sizing Materials* by J. H. MacGregor, *Felt* by T. Barr, *Chemical Testing* by F. F. Elsworth, and (a new section) *Industrial Applications of Textiles* by J. W. Illingworth. Presumably some of these titles will not appear every year.

At 35s. the review has become rather expensive, but, although some compression of the present volume could have been achieved by more severe editing, it is doubtful whether the price could have been reduced appreciably without some sacrifice of quality. How quality might be sacrificed can be seen if the contribution relating to the chemistry of cotton and cellulose in the current volume (pp. 25) is compared with that in the previous volume (pp. 7). Further support for this view is provided by the longest contribution in the current volume, viz. Mr. W. J. Marshall's excellent critical survey of the dyeing of textiles.

Our thanks are again due to all concerned with the production of what has become an essential tool in the promotion of textile technology.

C. L. Bird

A Handbook of Textile Finishing

By A. J. Hall. Pp. vii + 244. London; National Trade Press Ltd. 1952. Price, 25s. 0d.

The author of this book has chosen a simple and logical plan for the presentation of his subject-matter. After introductory chapters on the properties of the textile fibres, and yarns and fabrics, there follow chapters dealing with various finishing processes classified according to their ability to (*a*) modify appearances, (*b*) alter handle, (*c*) promote stability, and (*d*) improve serviceability of fabrics. The treatment on the whole is elementary, and the result is an extremely readable volume. The book will be of great service to beginners and those requiring a general introduction to finishing processes, but its use to serious students and research workers is much limited by the complete absence of literature references.

The 151 illustrations form a useful part of this book. The photographs of textile machines are clear and in general present a good view of the most important functional parts. A number of line diagrams illustrating the action of finishing machines or operations are also usefully included. Photographs of fibres, yarns, and fabrics illustrating special points in finishing are from authoritative sources. Chemical formulæ are, unfortunately, less well presented; a number of errors are to be noted, e.g. on pp. 67 and 180, and on p. 230 the description of Mitin FF does not fit the formula given. The reference to the use of acetone for resin formation (p. 194) should indicate that it may replace urea and not formaldehyde. Glyoxal is termed a "formaldehyde compound" on p. 232 when clearly *aldehydic* is meant.

On technical matters the author is generally sound, but it is doubtful, certainly in this country, whether glyoxal is preferred to formaldehyde for stabilisation of cellulosic fabrics. The impression that softening agents apparently reduce fabric strength is unfortunate, as in the reviewer's experience they may also have the reverse effect. The sticking temperature of nylon, given as 132° C., seems low (the du Pont *Technical Service Manual* gives 180° C.).

In addition to these points, it is surprising to find the names of the discoverers of our two most important synthetic fibres so mishandled. The

name of Carothers, of nylon fame, is misspelt throughout the book, and Cooke is given (p. 66) instead of Dickson as the codiscoverer with Whinfield of Terylene.

The price of the book is high, and can, perhaps, be justified only by the large number of illustrations included.

G. LANDELLS

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Clayton Aniline Co. Ltd.

CIBALAN DYES— This card contains dyeings on slubbing in six depths of twelve members of a new range of premetallised dyes. Unlike the earlier Neolan type, these are applied from neutral or very slightly acid baths to give dyeings having good fastness to light and wet treatments, including carbonising, peroxide bleaching, and chlorine. They also offer good resistance to a wide range of commercial non-shrink processes. They are recommended for dyeing loose wool and slubbing intended for carpet and hosiery yarns and for the manufacture of cloth for the ladies' and men's wear trade. In addition to fastness data, graphs are given for each dye illustrating the effect of temperature on dyebath exhaustion, and the rate of dyeing at 100°C., based on 1% dyeings and a 50 : 1 liquor : material ratio.

CIBANTINE BLUE 2G— This solubilised vat dye yields blues of good fastness to light, soda boiling, and chlorine on cellulosic fibres, and can be developed by both the chlorate–steam and nitrite processes. It is recommended for use alone or in mixtures for printing and pad dyeing, and is very suitable for coloured resists under aniline black and for pure white and coloured resists with vat dyes. Dyeings are readily dischargeable with a vat discharge paste. Fastness figures on cotton include— Light 6, chlorine 4, perspiration 5.

Farbenfabriken Bayer

DYEINGS ON JUTE YARN— This card (German text) contains 206 dyeings on jute produced with members of the following classes— acid dyes, including members of the Supranol, Supramine, and Acilan ranges; direct dyes, including Benzo, Benzo Fast, Sirius, and Sirius Supra dyes; and basic dyes, including six Janus dyes.

Farbwerke Hoechst

(In all cases the text is in German)

ANTHRACYANINE VIOLET 3R— This acid dye gives bright reddish violets of interest on wool, weighted and unweighted silk, and polyamide fibres of the Perlon and nylon types. Fastness figures on wool include— Light 4, water 4–5, washing 2.

WOOL FAST VIOLET FR— This acid dye is recommended for wool and silk and as a component of mixtures for dyeing mixtures of wool and cellulosic fibres. It is also of interest for direct printing of silk and wool. Fastness figures on wool include— Light 3, water 4, washing 2–3.

DYES FOR WOOL— This pattern card contains dyeings on wool cloth in two depths of thirty-seven acid dyes and fourteen chrome dyes applied by either the afterchrome or single-bath method.

ANTHRASOL PRINTING BLACK IGR— This is a solubilised vat dye recommended for application to cellulosic materials by either the chlorate–steam or the nitrite process to give prints of very good fastness to light and wet treatments. Fastness figures on cotton include— Light 7–8 (black print), chlorine 4–5, washing at 100°C. 4–5.

INDANTHREN PRINTING BLUE HFG— This vat dye is offered in Suprafix Paste form for direct printing and reserve styles in combination with dyes from the Anthrasol range. Pale shades may be discharged to a white using an alkaline Rongalite C–Leucotrope W discharge. Fastness figures on cotton include— Light 6–7, washing at 100°C. 4, chlorine 5.

INDANTHREN GREY HBR— This vat dye is recommended for use, alone and in mixtures, by the IW method on all types of cellulosic material where dyeings of good fastness to wet treatments are required. Type 8059 is suitable for pigment padding and the vat acid process. Fastness figures on cotton include— Light 6, washing at 100°C. 4, chlorine 4.

INDANTHREN BORDEAUX HRR— This vat dye may be applied by either the IN or the IN special method or pigment-padded, and is particularly suitable for use on furnishing fabrics and on yarn which is subsequently to be bleached in the piece. Fastness figures on cotton include— Light 6, soda boiling 3–4, chlorine 4–5.

INDANTHREN BRILLIANT GREEN H3G— This vat dye is supplied as a Suprafix Paste for direct printing on cotton, linen, regenerated cellulose rayon, and acetate rayon. Fastness figures on viscose rayon include— Light 7, chlorine 4–5, washing at 100°C. 5.

INDANTHREN COPPER HGR— This vat dye gives bright reddish browns of "Indanthren" fastness on all types of cellulosic materials. Fastness figures on cotton include— Light 6–7, washing at 100°C. 4, chlorine 5.

INDANTHREN BRILLIANT ORANGE RR— Bright reddish oranges of good fastness are produced by this vat dye when printed on cellulosic materials. Fastness figures on cotton include— Light 6, washing 4, chlorine 5.

HELINDONE TURQUOISE BLUE GF— This vat dye is intended for use alone and in combination with Helindone Brilliant Yellow GF to give pale greenish blues and greens of very good fastness to light and wet treatments on loose wool, slubbing, and cloth. Fastness figures include— Light 7, washing 5, potting 4, milling 5.

VAT PRINTING NAVY BLUE HR— A vat dye yielding reddish navy blues, recommended for printing all types of cellulosic material. Fastness figures on viscose rayon include— Light 5, washing at 100°C. 3–4, chlorine 5.

BRILLIANT CHROME LEATHER BLACK H— A direct dye for chrome-tanned leather.

BROWN HCG, HCM, HCD— These three direct dyes are recommended for producing light, medium, and dark reddish browns, respectively, on vegetable-tanned or synthetic-tanned leathers.

CHROME LEATHER BLACK HK SPECIAL— An acid dye suitable for use on all types of leather, particularly recommended for dyeing velour leather.

ACID LEATHER BROWN HEDG— An acid dye for use on chrome-, bark-, and synthetic-tanned leathers.

TANNING AGENTS, DYES, AND AUXILIARY PRODUCTS FOR THE LEATHER INDUSTRY— The selection and application of these products are outlined in this booklet.

Imperial Chemical Industries Ltd.

BEDAFIN 2001 AND BEDAFIN 285X— This pamphlet describes the properties, uses, and methods of application of these two synthetic-resin finishing agents.

THE SHIRLAN AND SHIRLAN NA BRANDS OF ANTI-MILDEW AGENTS— This pamphlet describes the properties, uses, and methods of application of these two products to cotton yarn and cloth; to made-up articles such as tents, bags, and saddles; and in the manufacture of paper and water paints.

THE PRINTING OF VISCOSE RAYON WITH ACID AND DIRECT COTTON DYESTUFFS (UREA PROCESS)— This card contains eighty prints on viscose rayon produced with thirty-two acid dyes, forty-seven direct dyes (including one two-component mixture), and two Naphthalene Leather dyes.

NAPHTHALENE LEATHER BROWN IT— This acid dye gives dull browns on a wide variety of leathers, the particular feature of which is the degree of penetration obtained together with full surface colour. This combination of properties is particularly desirable in grain leathers, since the "grinning" occurring when coloration is confined to the surface is here prevented by the presence of the

underlying dyed layer. Figures include— Light fastness 3, washing fastness 1, penetration 4, solubility 1.

DISPERSOL FAST YELLOW GS— This disperse dye gives bright greenish yellows on acetate rayon and nylon. It is particularly recommended for use on the latter fibre alone and in mixtures to give level dyeings of high light fastness and good wet fastness. Ground shades on acetate rayon are dischargeable, and the Printing Yellow GS Paste brand is suitable for the direct printing of both fibres. Fastness figures on nylon include— Light 6–7, washing (five times at 40°C.) 4, perspiration 3–4.

DYESTUFFS FOR "ARDIL" PROTEIN FIBRE–CELLULOSIC FIBRE UNIONS— This card contains sixty-nine dyeings arranged in two main groups— (1) On Ardil–viscose rayon mixture cloth, sixteen dyeings using acid dyes which leave the cellulosic fibre white or only slightly stained; twenty-one dyeings using selected direct dyes and dyeing conditions which leave the Ardil substantially undyed; seven solid dyeings using single direct dyes; and fourteen typical mixture dyeings using a single-bath process with acid dyes and direct dyes for the protein fibre and the cellulosic fibre, respectively. (2) On Ardil–cotton blended mixture yarn— thirteen solid shades dyed with single vat dyes or two-component mixtures.

DYESTUFFS FOR ARDIL PROTEIN FIBRE–WOOL CARPET YARNS— This card contains forty-five dyeings on wool–Ardil blended mixture yarn. These are mounted to show the cut ends of the fibres as is the case with a finished carpet. The dyeings include nineteen using single acid dyes and sixteen two- and three-component mixtures. The text includes notes on the additional factors which must be taken into account when dyeing this type of material, including the ground shade of the Ardil, the possible effect of formaldehyde, the effect of retained acid or alkali, and the wet fastness properties of the dyeings.

J. W. Leitch & Co. Ltd.

ORGANIC PIGMENTS AND LAKE COLOURS— This card shows fourteen pigments mixed with linseed oil in full strength and also reduced with zinc oxide in the proportion 1 : 10. A comprehensive list of fastness figures is appended.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index*

I— PLANT; MACHINERY; BUILDINGS

Vertical Screen Printing Machine. F. Zimmer. *Dyer*, **108**, 621–622 (31 Oct. 1952).

Screens are used vertically in pairs, clamping together over the cloth at the moment of printing; they register exactly with each other. Colour is applied by small rubber furnishing rollers (one to each side), each being aligned and supported by two rollers of larger diameter. This has the effect of rolling in colour from both sides simultaneously whilst the mutual pressure of the rollers expels air from the interstices; as the fabric expands it draws in colour and thus achieves a remarkable degree of penetration. Since the application of colour is by a rolling motion, and is lubricated by the colour itself, there is little wear on the screens, which have a life of ~ 100,000 impressions. Pressure is controlled by spring adjustment, and contrasting colours can be printed on opposite sides, meeting in the middle of the fabric. The cloth is automatically moved forward by one repeat after each impression, the length of repeat being easily set as required. The main novelty is that wide, thick materials may be printed on both sides, easily and economically, e.g. blankets, towels, quilts, and, possibly, carpets and floor-coverings. Narrow fabrics may be run side by side, and light ones back to back, to give doubled output. The first machine has been made to give repeats up to 100 in., but repeats up to 4 yd. are regarded as practicable. Numerous methods of printing thin fabrics (with which penetration is complete) and smaller repeats are described. Components such as screens and colour boxes are easily changed by hand, without tools, the cost of engraving screens is comparatively low, and since the machine is almost fully automatic, labour costs are very low. J. W. D.

Fundamental Problems of Drying by Radiation. G. Lück and C. Alt. *Allgem. Wärmetech.*, **3**, 58–66 (1952): *Chem. Abs.*, **46**, 9897 (10 Nov. 1952).

A theoretical discussion of the energy required, depth of penetration, and reflection of the radiation on the material. 16 references. C. O. C.

PATENTS

Stirring Dyebaths. L. Wyssling-Meier. *BP* 683,425

Air or other gas is fed to the bottom of the bath, and there distributed and liberated in a uniform manner. C. O. C.

Continuous Wet Processing of Textiles. Johnson & Johnson (Gt. Britain). *BP* 684,695

Wet processing of textiles is much more efficient and faster if the fabric, preferably in rope form, is advanced under tension through a tube full of the treating liquor, the fabric being fed into the tube so as to prevent voids in the cross-sectional area. C. O. C.

Mechanism for Controlling a Loop in a Web of Material. Sargrove Electronics. *BP* 684,234

A monitoring device responsive to the size of the loop, e.g. a system of lights and a photocell, controls the diameter of a feed roller, whose motion determines at least in part the size of the loop. C. O. C.

Impregnating Cord. B. F. Goodrich Co. *USP* 2,601,394

Simple means are described by which dry cord is impregnated with a colloidal dispersion by alternately untwisting and twisting the cord while immersed in the dispersion. C. O. C.

Register-producing Device for Textile or other Web-printing Machines. Joh. Kleinewefers Söhne. *BP* 684,277

A simple device made up of few parts and not needing a separate electric motor for the adjustment-effecting means. C. O. C.

Brushing Machines. J. F. A. Blue. *BP* 685,208

Providing the brushes with several helical and radially projecting flat strips with non-interrupted outer edges, each strip being fastened to the spindle of the brush and made from sponge, crêpe, or foam latex rubber, increases their efficiency. C. O. C.

Hosiery Press. A. Astphan. *BP* 682,957

Hosiery, e.g. men's socks, is placed on a row of individually pivoted, vertical, metal shapes which swing between heated plates to which pressure is applied. Each trio of plates along the machine operates as a single pressing unit on actuating the two outer plates. The operator is thus able to start the pressing operation on each pair as the forms are replenished. G. E. K.

Laundry Press. Brown & Green. *BP* 684,681

A press in which a sliding or smoothing action is effected. *BP* 684,682

An improved neckband block is described. C. O. C.

Facilitating the Threading of the Advancing End of the Paper Web through the Dryer. T. L. Coleman. *USP* 2,601,502

Pourers for Carboys. Harry Heymann. *BP* 685,163

A pourer having a curved air tube to project into the carboy, the tube being either made of an elastic material or mounted elastically so that during pouring its inner end is kept against the inside wall of the carboy, thus preventing liquid in the carboy from getting into the air tube at any time during pouring. C. O. C.

Sonic Vibration as an Aid in Dyeing (VIII p. 103).

II—WATER AND EFFLUENTS

Practical Approach to Textile Pollution Abatement and Waste Treatment. D. W. Snyder. *Amer. Dyestuff Rep.*, **41**, 745–746 (10 Nov. 1952).

A brief survey of pollution sources and the remedies. J. W. B.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Ethylene Oxide Derivatives and their Uses in the Textile Industry. G. Trinchieri. *Tinctoria*, **48**, 132–135 (May), 171–174 (June 1951): *Amer. Dyestuff Rep.*, **41**, 729–732, 751–752 (10 Nov. 1952).

Ethylene oxide derivatives are divided into those not having and those which have surface-active properties, the latter taking up most of the survey, which covers their use as oil-soluble emulsifying agents, wetting agents, and detergents, softeners, and dyeing assistants. The influence of chain length on wetting, dispersing, and detergent properties is briefly discussed, and data are given on comparative wetting properties, under various conditions, of different types of agent. J. W. B.

Autodecomposition of Hydrogen Peroxide. E. Abel. *Monatsh.*, **83**, 422–439 (1952): *Chem. Abs.*, **46**, 10,812 (25 Nov. 1952).

Autodecomposition of H_2O_2 has the following mechanism—

(i) $H_2O_2 \rightleftharpoons HO_2^- + H^+$

(ii) $HO_2^- + H_2O_2 \rightarrow HO_2 + OH^- + OH$ (rate-determining step)

(iii) $HO_2 + OH \rightarrow H_2O + O_2$

Kinetic equations are given for this mechanism. The reaction speed passes through a maximum with increasing alkali addition. Considerable parallelism exists between the uncatalysed and metal–colloid-catalysed reactions, and it seems that the same mechanism applies to both, the sole function of the metal catalyst being to facilitate electron transfer between HO_2^- and H_2O_2. C. O. C.

Mechanism of the Decomposition of Hydrogen Peroxide in Certain Solid Perhydrates. I. A. Kazarnovsky and A. B. Neyding. *Doklady Akad. Nauk S.S.S.R.*, **86**, 717–720 (1 Oct. 1952).

Potassium peroxide perhydrates of composition varying from $K_2O_2,4{\cdot}5H_2O_2$ to $K_2O_2,2H_2O_2$ are prepared by evaporating a soln. containing two moles of H_2O_2 and one mole of KOH over conc. H_2SO_4 *in vacuo*; further exposure of $K_2O_2,2H_2O_2$ to this treatment leads to formation of yellow KO_3. The decomp. of the perhydrates is followed by measuring the oxygen evolved and by determining the content of KO_3, which is paramagnetic, by magnetic susceptibility measurements. In the decomposition of $K_2O_2,2H_2O_2$, KO_3 and O_2 are produced, at first at const. rate in about equimolecular proportions. Later, the amount of KO_3 present falls slowly, while oxygen evolution continues at a very low rate. If the reaction is carried out *in vacuo* over a drying agent, oxygen evolution is largely suppressed, 91% conversion to KO_3 being obtained, and it is clear that the fundamental reaction is—

$$K_2O_2,2H_2O_2 = 2KO_3 + 2H_2O$$

The mechanism proposed for this reaction is considered to throw light on the mechanism of the decomp. of H_2O_2 in alkaline soln., which is considered to be as follows—

$$O_3^{2-} + H_2O_2 = O_3^{\cdot-} + OH^- + OH$$
$$(\text{or } HO_2^- + H_2O_2 = HO_2 + OH^- + OH)$$
(Initiation of Chain)

$$H_2O_2 + OH = H_2O + HO_2$$
$$HO_2 = H^+ + O_2^-$$
$$H_2O_2 + O_2^- = OH^- + O_2 + OH$$
(Chain Reaction)

The function of the alkali is to increase the electrolytic dissociation of H_2O_2. A. E. S.

Manufacture of Sodium Hydrosulphite (Dithionite). T. Dijs, J. G. Hoogland, H. I. Waterman, J. J. G. Wahlen, and A. L. Mahler. *Chem. and Ind.*, 1073–1075 (1 Nov. 1952).

Dithionite is manufactured technically by reaction of gaseous SO_2 with a suspension of Zn dust in water with final addition of NaOH—

$$Zn + 2SO_2 \longrightarrow ZnS_2O_4$$
$$ZnS_2O_4 + 2NaOH \longrightarrow Zn(OH)_2\downarrow + Na_2S_2O_4$$

This process is described in *BIOS* 422, and gives a yield of 72%. Another practicable but more costly process is the cathodic reduction of $NaHSO_3$—

$$2HSO_3^- + 2\ominus + 2H^+ \longrightarrow S_2O_4^{2-} + 2H_2O$$

This process is patented (*USP* 2,193,323), and though the inventor claims a yield of 90%, the authors obtained only 75%, attributing this to possible deficiencies in their plant. Suitable pilot plant for cathodic reduction is described in *BIOS* 1373. The authors give details of their investigation of a third process (which is described also in the above report), viz. reaction between SO_2 and sodium amalgam; an earlier investigator found this to give a yield of 50–70%, this condemning it economically. The present authors find that, if certain conditions are maintained in the reactor, the yield loss of 20% previously reported can be materially reduced. Two types of reactor are described and compared. An advantage of this method is that it eliminates the filtration of $Zn(OH)_2$ necessary with the Zn dust process. The choice of process depends largely on the prices of Zn dust and ZnO (by-product); in Holland, the country of origin of the authors, the balance is in favour of the amalgam process. J. W. D.

Evidence of Multiple Branching in Waxy Maize Starch. S. Peat, W. J. Whelan, and G. J. Thomas. *J.C.S.*, 4546–4548 (Nov. 1952).

Synthesis of "Organo-elementary" Compounds of the Aliphatic Series by the Diazo Method. III—Compounds of Group IV Elements—Organosilicon Compounds. A. Ya. Yakubovich and V. A. Ginsburg. *J. Gen. Chem. U.S.S.R.*, **22**, 1783–1787 (Oct. 1952).

$SiCl_4$ and $SiBr_4$, but not SiF_4, react with diazomethane or diazoethane at $\sim -50°$C. to give mono-, bis-, or tris-α-halogenoalkylsilicon halides, e.g.—

$$SiCl_4 + CH_2N_2 = CH_2Cl\text{-}SiCl_3 + N_2 \ (\sim 70\% \text{ yield})$$

Alkylsilicon halides react with difficulty, and the trialkylsilicon halide examined, $(C_2H_5)_3SiCl$, failed to give the reaction. Copper or copper salts may be used as catalysts. A. E. S.

PATENTS

2-Pyridyltrimethylsilane—Emulsifying and Ion-exchange Agent. British Thomson-Houston Co. *BP* 685,186

2-Pyridyltrimethylsilane is a useful intermediate in making other organic compounds. Some of its salts are useful as emulsifying agents, ion-exchange agents, etc. C. O. C.

Detergent. General Aniline. *USP* 2,601,329

Polyglycol ethers of alkylphenols and similar detergents do not discolour even on prolonged storage in contact with caustic alkalis if about 10% by wt. of thiourea is incorporated. C. O. C.

Soap Powder resistant to Hard Water. Lever Brothers & Unilever. *BP* 685,049

Soap powder made from molten soap and soda ash and then mixed with $\geqslant$ 15% by weight of finely divided $CaCO_3$ quickly softens hard water without formation of calcium soap. C. O. C.

Alkarylsulphonate–Hydroxyethyl Cellulose Detergents. Wyandotte Chemicals Corpn. *USP* 2,602,781

Addition of hydroxyethyl cellulose to alkarylsulphonates much increases their detergent power; e.g. addition of 5–57% hydroxyethyl cellulose to sodium kerylbenzenesulphonate increases its detergent properties by 50–90%. C. O. C.

Modified Urea–Formaldehyde Condensates having Affinity for Cellulose. British Industrial Plastics. *BP* 684,091

Water-soluble cationic condensates substantive to cellulose are obtained by boiling 1·5–3 mol. of formaldehyde at pH 3·5–6·0 with 1 mol. of urea in aqueous soln. in presence of a dicyandiamide–formaldehyde acid condensate (so that 3–100 mol. of dicyandiamide are used per 100 mol. of urea) and neutralising. They are used *inter alia* for finishing textiles and increasing the wet strength of paper. C. O. C.

Urea–Melamine–Formaldehyde Resins. ICI. *BP* 683,630

Stable resin syrups suitable for impregnating textiles are obtained by treating a mixture of melamine (65–95% by wt.) and urea (35–5) with formalin at pH 3–10 until the

condensate has a water tolerance of $\leqslant$ 3 : 1 (i.e. vol. of water needed to render the clear resin syrup opalescent). There should be $\leqslant$ 2 mol. of formaldehyde for each mol. of urea plus $\leqslant$ 3 mol. of formaldehyde for each mol. of melamine. The 10–40% of water on the wt. of the condensate is then removed by dehydration under vacuum followed by dilution to the desired resin content with an alcohol of < 6 C. C. O. C.

Stable Water-soluble Urea–Aldehyde Condensates. Deutsche Gold- und Silber-Scheideanstalt vormals Roessler. *USP* 2,600,780

Urea–aldehyde condensates produced by reaction of urea and/or thiourea with acrolein under acid conditions have a high peptising effect, so that when the products are subsequently treated with formaldehyde in presence of basic catalysts the end products form solutions free from precipitates, miscible with water in all proportions, and of unlimited stability. C. O. C.

Polymers of αβ-Unsaturated Carboxylic Acids— Auxiliary Agents. Henkel & Cie. *BP* 685,151

The products obtained by heating and agitating, in presence of polymerisation accelerators and in absence of molecular oxygen, the mixtures obtained by the strong mineral acid hydrolysis of ketone cyanohydrins are useful as thickening agents, protective colloids, dyeing assistants, and colour-binding agents. C. O. C.

Water-soluble Derivatives of Lignin— Tanning Agents. DuP. *USP* 2,600,606

Excellent tanning agents are obtained by treating lignin with boric acid and phenols, preferably polyphenols, and then neutralising. C. O. C.

Protein Hydrolysis Product. Union Starch & Refining Co. *BP* 685,376

Purified gluten (obtained by treating commercial gluten to free it from starch and starch degradation products) is hydrolysed in a closed vessel with an aqueous suspension of lime at pH 9–13·5 and 115–135°c., using either external heat or internal steam. Undissolved protein is removed, the remainder acidified to neutralise the lime, and the product separated. It can be added to leather products as a filler, being pptd. from aq. soln. by tannins; or if made in water-insoluble form, it is soluble in aq. alcohols and dil. aq. Na_2CO_3 or ammonia, and is adherent to glass, being useful e.g. for back-coating photographic plates. The processes are summarised in a concise flow-diagram. J. W. B.

Water-in-lacquer Emulsions for Application to Textiles (IX p. 103).

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Bromination with Dioxan Dibromide. I— Bromination of Phenols. L. A. Yanovskaya, A. P. Terentyev, and L. I. Belenky. *J. Gen. Chem. U.S.S.R.*, **22**, 1594–1598 (Sept. 1952).

The addition product formed when one mole of bromine is added to 1 mole of dioxan is a useful brominating agent for phenols, since it permits the preparation of mono- or di-bromo derivatives that are otherwise difficult to prepare. The monobromination of phenol, anisole, thymol, 1:3:5-xylenol, catechol, orcinol, guaiacol, α- and β-naphthols, β-methoxynaphthalene, α- and β-hydroxyanthraquinones, alizarin, salicylic acid, and *p*-nitrophenol, and the dibromination of quinol, resorcinol, pyrogallol, and phloroglucinol, are described. A. E. S.

Mechanism of the Reaction of Nitrogen Dioxide with Aromatic Hydrocarbons. A. I. Titov. *J. Gen. Chem. U.S.S.R.*, **22**, 1329–1335; **Mechanism of the Nitration of Aromatic Compounds with Nitric Acid. III— Nitration of Benzene with Diluted Nitric Acid.** A. I. Titov and A. N. Baryshnikova. *Ibid.*, **22**, 1335–1338 (Aug. 1952).

Titov has previously established (cf. *Chem. Abs.*, **42**, 545*i* (1948)) that, in the nitration of organic compounds by means of nitric acid of less than a certain concn. that is characteristic for each cpd., the effective nitrating agent is the radical-like NO_2 molecule, which in a non-polar medium forms an intermediate addition cpd. with the aromatic substance. Further reactions, the mechanism of which is discussed, can lead to the formation of polynitro derivatives and nitrophenols. The reaction between benzene and NO_2 is studied at ordinary temp. and pressure and also in closed tubes at 100–150°c. It is favoured by dilution of the mixture with a hydrocarbon solvent, and the products are nitrobenzene, *m*- and *p*-dinitrobenzene, *s*-trinitrobenzene, 2:4-dinitrophenol, 2:4:6-trinitrophenol, and oxalic acid. When toluene or chlorobenzene is used in place of benzene, the trinitro derivatives of *m*-cresol or of *m*-chlorophenol respectively are the chief products. Nitration of benzene with nitric acid of sp. gr. < 1·2 proceeds almost entirely via the intermediate reaction with NO_2, and, in accordance with the authors' theory, the main final products are *s*-trinitrobenzene, picric acid, and nitrobenzene, which are formed in almost equal quantities. On increasing the concn. of HNO_3, the yield of nitrobenzene increases, since it is then formed by the normal nitration mechanism, which does not involve the participation of the neutral NO_2 molecule. A. E. S.

Reaction of Sulphonation. XXIII— Preparation of the Acid Chlorides and other Derivatives of Naphtholdisulphonic Acids. A. A. Spryskov and N. V. Aparyeva. *J. Gen. Chem. U.S.S.R.*, **22**, 1624–1631 (Sept. 1952).

The reactions between chlorosulphonic acid and 1:3:8-, 1:4:8-, 2:6:8-, and 2:3:6-naphtholdisulphonic acids are studied in some detail. The naphthosultone-3- and -4-sulphonyl chlorides that result from reaction with 1:3:8- and 1:4:8-naphtholdisulphonic acids respectively are converted into the hitherto uncharacterised diamides and dianilides of the original disulphonic acids and into the anilides of the naphthosultonemonosulphonic acids. In contrast to the 2:6:8 acid, which gives the expected dichloride with chlorosulphonic acid, the 2:3:6 acid (R acid) gives 2-naphthol-1:3:6-trisulphonyl chloride. A. E. S.

Reaction of Sulphonation. XXIV— Hydrolysis of Naphthalenedisulphonic Acids. A. A. Spryskov and B. I. Karavaev. *J. Gen. Chem. U.S.S.R.*, **22**, 1871–1875 (Oct. 1952).

The six naphthalenedisulphonic acids formed in the direct sulphonation of naphthalene are hydrolysed by water and by sulphuric acid (35–65%) at 100, 124, and 161°c. With respect to their resistance to hydrolysis, the acids form the following series— 2:6 > 2:7 > 1:3 > 1:7 > 1:6 > 1:5. Contrary to indications in the literature, the two sulpho groups do not split off simultaneously— the monosulphonic acid is formed first, and is then hydrolysed to naphthalene. A. E. S.

Preparation of some 4-*n*-Alkyl-3-nitrobenzoic Acids. H. A. Fahim and A. M. Fleifel. *J.C.S.*, 4519–4521 (Nov. 1952).

p-Alkylbenzoic acids are prepared by alkaline fusion of the corresponding *p*-alkylphenyl methyl ketones at 200–280°c., and are best nitrated by fuming nitric acid alone or by a mixture of conc. HNO_3 and conc. H_2SO_4. H. H. H.

Crystallographic Data on 2:4-Dinitrophenol. W. C. McCrone and J. Kre. *Anal. Chem.*, **24**, 1863–1864 (Nov. 1952).

Action of Nitrous Acid on 2-Methoxydimethylaniline. N. N. Sveshnikov, I. I. Levkoev, and T. V. Krasnova. *J. Gen. Chem. U.S.S.R.*, **22**, 1170–1172 (July 1952).

Contrary to statements in the literature (Grimaux and Lefèvre, *C.R. Acad. Sci. Paris*, **112**, 727 (1891)), 2-methoxydimethylaniline reacts like various alkyldimethylanilines with nitrous acid, yielding not 2-methoxy-4-nitrodimethylaniline alone, but a mixture of this substance with 2-methoxy-4-nitrophenylmethylnitrosamine. A. E. S.

Preferential Reduction of Nitro Groups in Polynitro Compounds. V— 1:3:6-, 1:3:8-, and 1:4:5-Trinitronaphthalene. E. R. Ward and L. A. Day. *J.C.S.*, 4529–4530 (Nov. 1952).

A 25% yield of 3:6-dinitro-1-naphthylamine results from the $SnCl_2$–HCl reduction of 1:3:6-trinitronaphthalene, but the main reaction with NaSH appears to be replacement of one or more NO_2 groups by sulphur-containing radicals. No successful $SnCl_2$–HCl reduction can be reported for 1:3:8-trinitronaphthalene, while 1:4:5-trinitronaphthalene appears to afford a mixture of two amines, and with NaSH the main product appears to be sodium 4:8-dinitro-1-naphthyl sulphide. 1:4:5:8-Tetranitronaphthalene gives

4:8-dinitro-1:5-di-*o*-nitrophenylthionaphthalene when treated with sodium *o*-nitrothiophenoxide. H. H. H.

Scope of the Knoevenagel Synthesis of Aromatic Secondary Amines. Ng. Ph. Buu-Hoï. *J.C.S.*, 4346–4349 (Nov. 1952).

Knoevenagel's method for preparing aromatic secondary amines, by iodine-catalysed condensation of hydroxy compounds with aromatic amines, is extended, as regards the phenolic component, to several polyhydric benzene and naphthalene derivatives and to 6-bromo-2-naphthol; and, as regards the amino component, to a series of substituted anilines and to 2-aminopyridine. Attempts to synthesise *N-cyclo*alkyl-β-naphthylamines resulted chiefly in di-β-naphthylamine. This synthesis can be extended to the heterocyclic group. H. H. H.

Study of Diazo Compounds by use of Nitrogen Isotopes. Reduction of Mixed Diazonium Salts of Anthranilic Acid and Aniline. P. F. Holt and B. I. Hopson-Hill. *J.C.S.*, 4251–4254 (Nov. 1952).

Benzene- and *o*-carboxybenzene-diazonium chlorides when reduced separately by the cuprammonium ion give azobenzene and diphenic acid respectively. When the mixed solutions are so reduced, azobenzene-2-carboxylic acid is formed in addition, and its nitrogen is found to be derived not from the benzene— but from the *o*-carboxybenzene-diazonium radical. H. H. H.

Effect of Methylation on the Light Absorption of Phenols—I. A. Burawoy and J. T. Chamberlain. *J.C.S.*, 2310–2316 (June 1952).

The spectra of numerous substituted phenols and of the corresponding anisoles in ethyl alcohol are compared. Methylation displaces both the *B*- and the *K*-bands to shorter wavelengths and reduces their intensity. If both *ortho* positions to O·CH_3 are occupied by Cl, Br, I, CH_3, CHO, or NO_2, this effect is considerably enhanced owing to the intramolecular steric hindrance between the O·CH_3 and one of the substituents. The cause of the steric inhibition of conjugation is briefly discussed. H. H. H.

Effect of Methylation on the Light Absorption of Phenols. II— Monophenylazophenols. A. Burawoy and J. T. Chamberlain. *J.C.S.*, 3734–3740 (Oct. 1952).

The spectra of 4-methyl-2-phenylazophenol, 4-phenylazophenol, and 4-phenylazoresorcinol, as well as of 2- and 4-nitrophenol and 4-nitroresorcinol, are compared with those of their methyl ethers. In absence of hydrogen-bond formation and steric effects, the normal effect of methylation (cf. Part I, preceding abstract) is a displacement of *B*- and *K*-bands to longer wavelengths. Displacements to shorter wavelengths may be due to a reversal of the effect of hydrogen-bond formation and also, in phenols substituted in the *ortho* position, to reduced conjugation caused by intramolecular steric effects. The electronic spectra can serve as a test for the presence of an internal hydrogen bond in phenolic substances. The *o*-hydroxyazo compounds investigated exist in solution as true phenols. H. H. H.

Spectroscopic Investigation of Orange I and Orange II. J. B. Müller, L. Blangey, and H. E. Fierz-David. *Helv. Chim. Acta*, **35**, 2579–2589 (Dec. 1952).

By comparison of the absorption spectra of Orange I and its *N*-methyl and *O*-methyl deriv. it was shown that in neutral aq. soln. Orange I exists in the quinone hydrazone form. It was also demonstrated that the singly charged anion of Orange II is very largely in the quinone hydrazone state. In alkaline soln., Orange I and II correspond neither to the azo nor to the hydrazone form, but are doubly charged, mesomeric anions. The effect of addition of alkali on the spectra of both cpd. was investigated, and their p*K* (8·1 and 11·4) were determined. Orange II was *O*-alkylated with dimethyl sulphate in conc. aq. NaOH at 90°c. H. E. N.

Preparation of Symmetrical Dibromo-, Dinitro-, and Dihydroxy - azonaphthalenes. B. M. Bogoslovsky and Z. S. Kazakova. *J. Gen. Chem. U.S.S.R.*, **22**, 1183–1186 (July 1952).

In a previous paper (cf. *Chem. Abs.*, **41**, 104*i* (10 Jan. 1947)) it was shown that, in the Sandmeyer reaction, increase in rate of adding diazo soln. to the Cu salt leads to formation of symmetrical azo compounds, e.g. *oo'*-azotoluene from diazotised *o*-toluidine. A method was devised on this basis, using a soln. prepared by reducing $CuSO_4$ in ammoniacal soln. with hydroxylamine, for the preparation of such azo compounds in good yield, and it was applied mainly to the preparation of simple derivatives of azobenzene. It is now further applied to the preparation in good yield of symmetrical derivatives of the azonaphthalenes, viz. 1:1′-dibromo-2:2′-azo-, 4:4′-dibromo-1:1′-azo-, 5:5′-dibromo-1:1′-azo-, 5:5′-dinitro-1:1′-azo, 4:4′-dinitro-1:1′-azo-, 5:5′-dihydroxy-1:1′-azo-, and 7:7′-dihydroxy-1:1′-azo-naphthalene. 8-Nitro-1-naphthylamine and 1-nitro-2-naphthylamine do not give the expected azo compounds, but yield, instead, the corresponding symmetrical dinaphthyls. A. E. S.

Preparation and Reductive Fission of Alkyl Ethers of Phenylazonaphthols. J. B. Müller, L. Blangey, and H. E. Fierz-David. *Helv. Chim. Acta*, **35**, 2574–2579 (Dec. 1952).

By a variation of previous methods, 1-phenylazo-2-naphthol (I) and 2-phenylazo-1-naphthol were converted into their methyl (at 75°c.) and ethyl (at 100–105°c.) ethers using 2 mol. of dialkyl sulphate. 1-Phenylazo-4-naphthol required only 1·5 mol. of alkylating agent. Catalytic reduction of these ethers afforded the corresponding aminonaphthyl alkyl ethers and aniline quantitatively. Reduction of the methyl ether of I with $SnCl_2$ in aq. HCl gave, besides normal products, 24% of 4-amino-3-methoxy-1-*p*-aminophenylnaphthalene. H. E. N.

Researches on Monolayers. III— Effect of Mono- and Di-hydric Phenols on Monolayers of Certain Aromatic Compounds. C. H. Giles and E. L. Neustädter. *J.C.S.*, 3806–3813 (Oct. 1952).

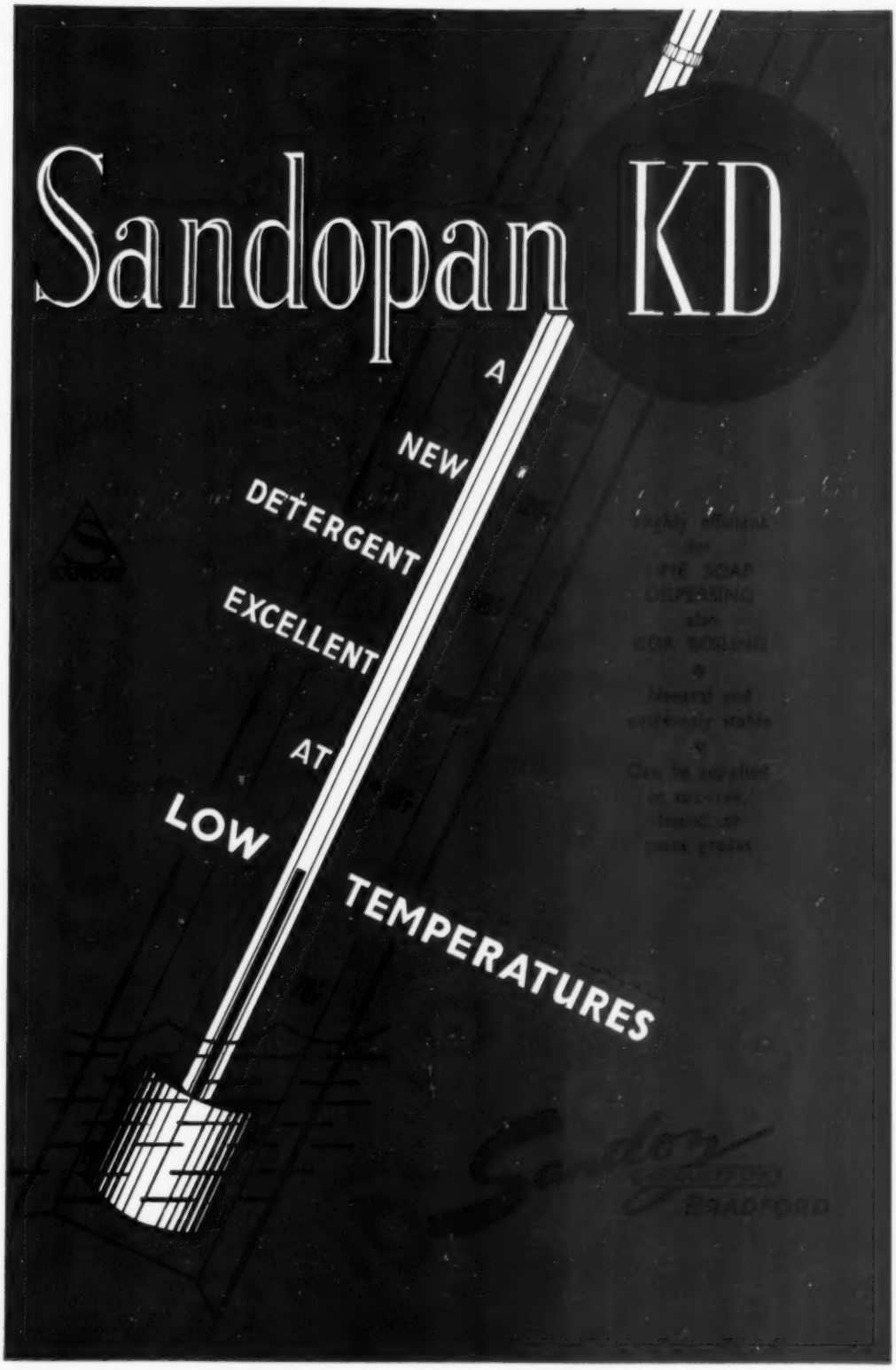
Azo, hydroxy, and quinone groups in monolayers of aromatic compounds on water form a hydrogen bond with the hydroxy group in monohydric phenols dissolved in the aqueous phase. The increased water attraction thus imparted, e.g. to the azo group, may cause an expansion of the film by a change in the angle of tilt of the film molecule. Dihydric phenols in the water act in two ways— (*a*) If there are two suitably placed bonding groups in the monolayer molecule a 1 : 1 molecular complex may be formed, in which each of these groups is bonded with one hydroxy group in the solute molecule. Quinol acts thus on (i) some hydroxyazo compounds and (ii) benzoquinone and anthraquinone derivatives; with (ii) a quinhydrone-type compound appears to be formed. (*b*) If the solute hydroxy groups are too far apart for (*a*) to occur, they may form cross-links between monolayer molecules, leading to a considerable increase in film area and compressibility. That the observed effects are due to hydrogen bonding, and not merely to penetration of the film by bulky solute molecules, is shown by the absence of significant changes in film area or compressibility when pyridine is used as solute below films of compounds with which it cannot form a hydrogen bond. H. H. H.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XI— Oxidation Potentials of Naphthidine, 3:3′-Dimethylnaphthidine, and their Sulphonic Acids. R. Belcher, A. J. Nutten, and W. I. Stephen. *J.C.S.*, 3857–3861 (Oct. 1952).

Transition potentials of naphthidine, naphthidine-sulphonic acid, 3:3′-dimethylnaphthidine, and 3:3′-dimethylnaphthidinedisulphonic acid, determined by comparison with the molar electrode potentials of the ferrous–ferric and dichromate–chromic systems in various concentrations of acid, are 0·800, 0·840, 0·706, and 0·800 volt respectively in M-H_2SO_4. Consideration of the structure of the indicator molecules shows that sulphonation, as with diphenylamine (transition potential 0·760 v.), leads to an increase in oxidation potential, and the presence of methyl groups in the naphthidine nucleus reduces the potential considerably. The results obtained from the direct titration of the indicators with ceric sulphate show this method to be of little value for determination of the oxidation potentials. H. H. H.

Internuclear Cyclisation. VI— Synthesis of some Methyl-, Halogeno-, and Carbomethoxy-*N*-methylphenanthridones. R. A. Heacock and D. H. Hey. *J.C.S.*, 4059–4064 (Oct. 1952).

The preparations of 2-amino-*N*-methylbenzo-*p*- and -*o*-toluidide and of 2-amino-4′-bromo-, 2-amino-4′-chloro-,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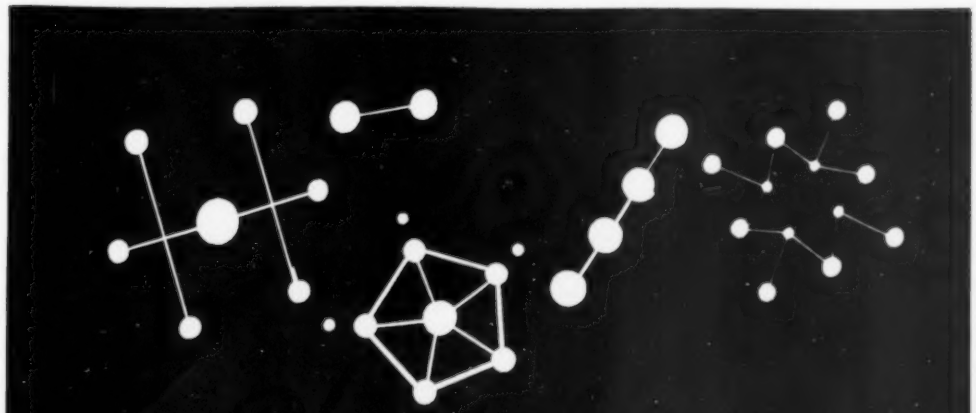

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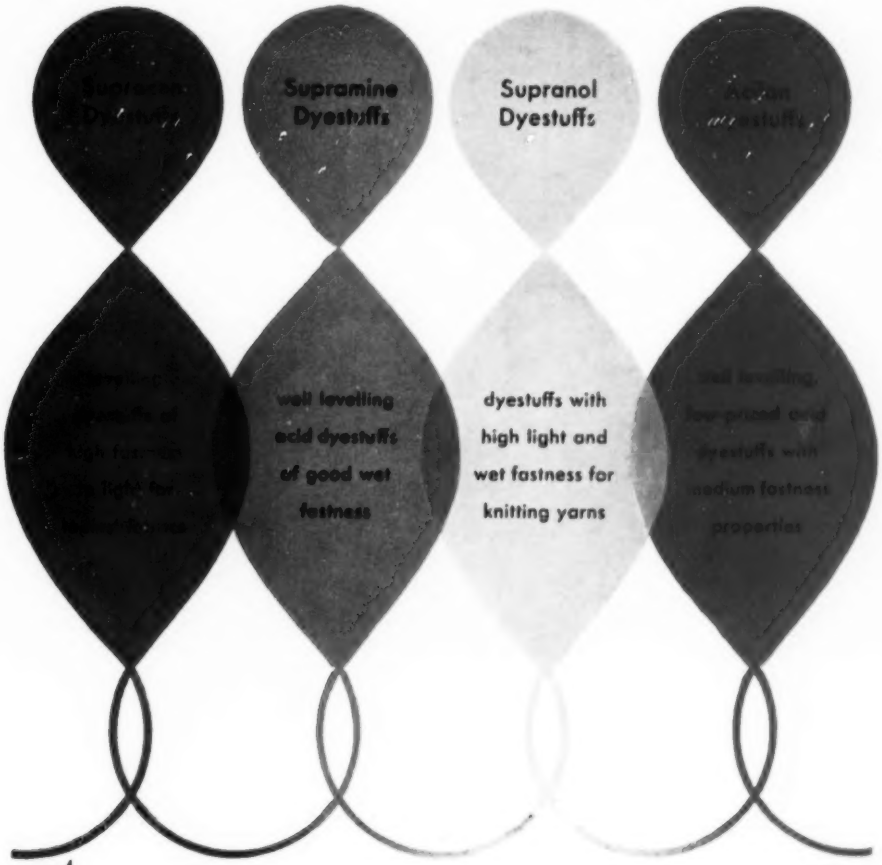
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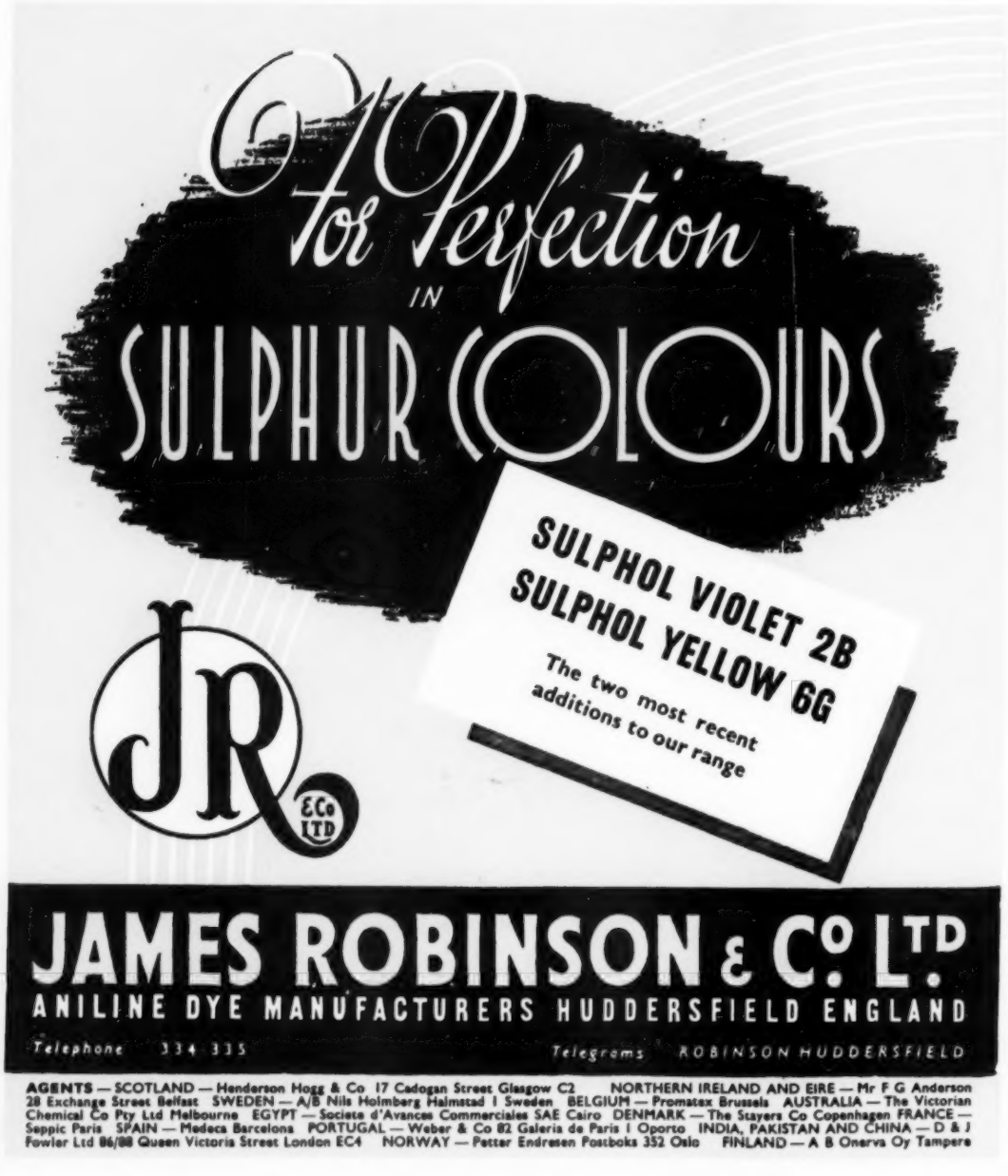

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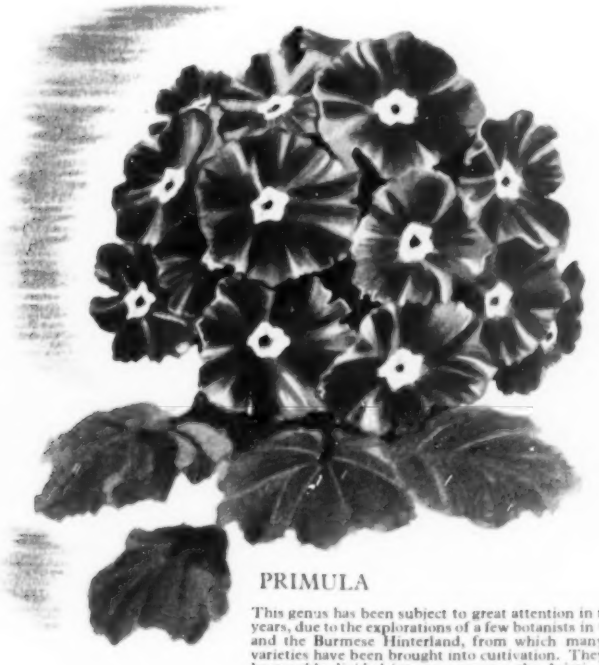

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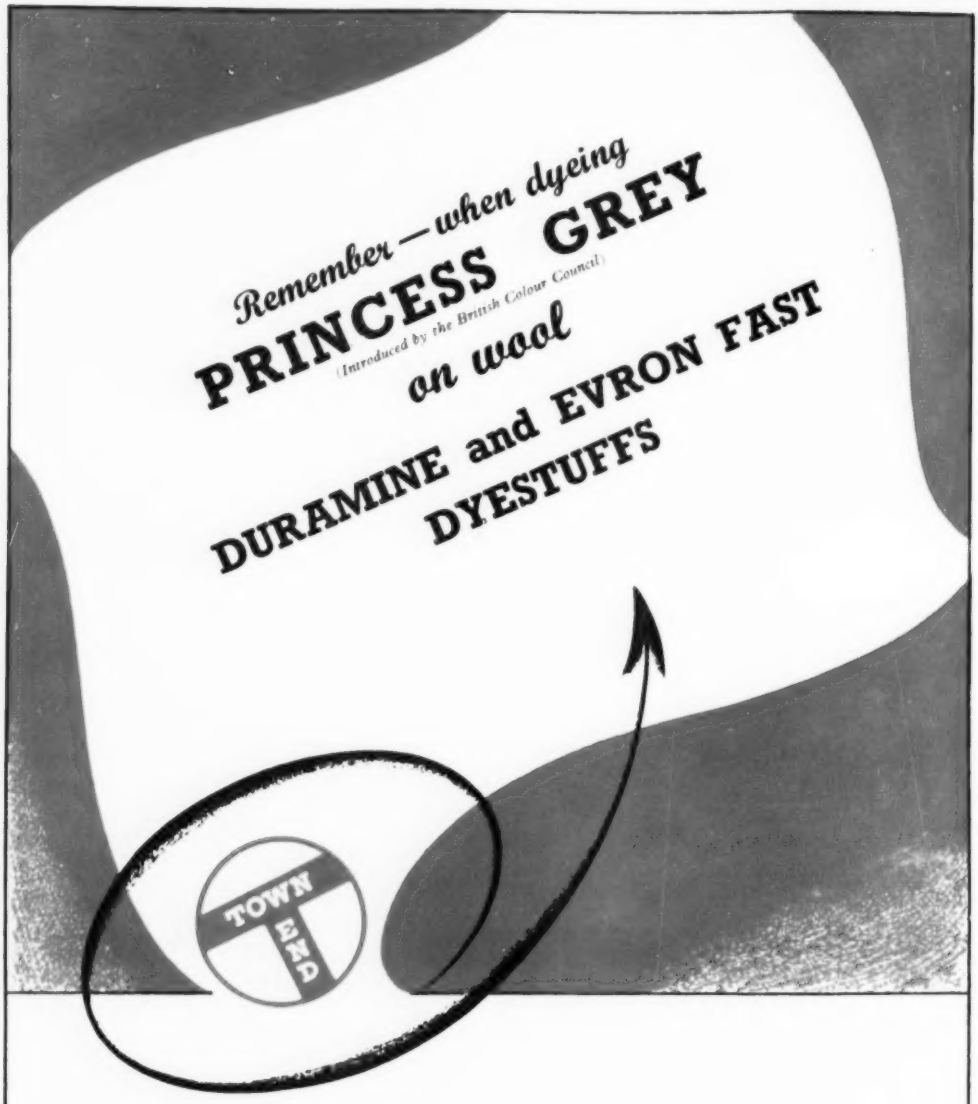

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2-amino-2′-chloro-, and 2-amino-4-carbomethoxy-*N*-methylbenzanilide are described, together with a study of the decompositions of their diazonium sulphates in aqueous solution and of their diazonium fluoroborates in acetone suspension. Substituted *N*-methylphenanthridones are obtained in each case except with 2-amino-2′-chloro-*N*-methylbenzanilide, which affords unidentified substances. The corresponding phenols accompany the *N*-methylphenanthridones from the decomposition of the diazonium sulphates. H. H. H.

Contributions to Dye Chemistry by François-Zacharie Roussin. E. Justin-Mueller. *Teintex*, **17**, 577–581 (Sept. 1952).

A record of the work of F.-Z. Roussin (1827–1894) with special reference to organic chemistry and dyes. B. K.

The Chelate Effect. G. Schwarzenbach. *Helv. Chim. Acta*, **35**, 2344–2359 (Dec. 1952).

The chelate effect is defined as the stability difference between a chelate complex and the corresponding complex with simple ligands. Equations are derived for the prediction of its magnitude. These are discussed for three types of chelate ring— (1) small and free from strain, (2) large, and (3) strained. In the first case, it is shown that the chelate effect is largely independent of the strength of the metal–ligand bond, because it is an entgopy effect. Complexes with polydentate reagents are also considered. H. E. N.

Structures and Properties of Some Metal Derivatives of Azo and Azomethin Dyes. J. C. Bailar and C. F. Callis. *J. Amer. Chem. Soc.*, **74**, 6018–6021 (5 Dec. 1952).

Study of the Co lakes of phenylazo-β-naphthol and of the Co, Cr, and Zn lakes of 2′-hydroxybenzylidene-2-hydroxy-5-chloroaniline indicates that they can be fitted into a general pattern that permits prediction of the types of azo dye complexes that certain metals may be expected to form. The oxidation state of the Co was + 3 in the mono*ortho* dye lakes and either + 2 or + 3 in the di*ortho* dye lakes. The lakes of tervalent Co can be prepared by replacement reactions using Co(III) ammine complexes as source of tervalent Co. The lakes of bivalent Co and Zn are analogous in composition, as are those of tervalent Co and Cr. It seems that a mono*ortho*-substituted azo dye can satisfy one primary valence and two secondary valences of the metal atom. Di*ortho*-substituted dyes can satisfy three secondary valences and two primary valences of the metal atom. C. O. C.

Effect of the Anion on the Conductive Properties of Triphenylmethane Dye Salts. R. C. Nelson. *J. Chem. Phys.*, **20**, 1327–1328 (1952): *Chem. Abs.*, **46**, 10,860 (25 Nov. 1952).

Crystal violet bisulphate and methyl violet bisulphate show the same order of dark resistivity as the chlorides, but show very much less photoeffect. The sulphates and oxalates of these dyes are very strongly photoconductive in visible light and also have a low dark resistivity. It is thought that the conduction band is an extended mutual excited state of the dye cations. On this basis the anion becomes a donor "impurity" level, and the primary process is the thermal excitation of an electron to the conduction band. C. O. C.

Kinetics of the Formation of the Carbinol of Crystal Violet. J. C. Turgeon and V. K. LaMer. *J. Amer. Chem. Soc.*, **74**, 5988–5995 (5 Dec. 1952).

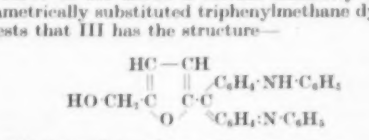
Fading of crystal violet in presence of hydroxyl ion follows quantitatively the Bronsted–Debye law for primary kinetic salt effects. Crystal violet obeys Beer's law at the wavelength of maximum absorption (590 mμ.) in concentrations $> 5 \times 10^{-6}$ M. The activation energy for the reaction in water at 25°c. is 15·1 kg.cal./mole; entropy of activation under these conditions is − 12·3 cal./°c. per mole. There is a strong specific solvent effect: addition of acetone or dioxan to an aq. soln. of the dye produces shift in the absorption peak of the crystal violet ion toward longer wavelengths and, at low concentrations of organic solvents, produces an effect on the specific reaction rate which is opposite to that expected on the basis of the decrease in dielectric constant. Use of normalised rate constants does not improve the agreement with simple electrostatic theory. Energy of activation in 40% acetone–water mixtures (dielectric constant 59) at an ionic strength of 0·005 is 0·9 kg.cal./mole greater than with water alone at this ionic strength, contrary to the expected decrease due to the lowering of the coulombic activation energy in this reaction between oppositely charged ions produced by lowering the dielectric constant. Activation energy for carbinol formation of malachite green is 0·7 kg.cal./mole less than that for crystal violet. The higher rate constant of the former (2·29 as against 0·25 litre/mole–sec.) is due to reduction in activation energy, also to a less negative entropy of activation. The gas kinetic collision theory agrees with the observed rate for a probability factor $P = 0{\cdot}098$ for $(r_a + r_b) = a_i = 2{\cdot}49$ A. P for the Debye theory (frequency of encounter, based on diffusion theory) is 0·84 for the reaction in water and 3·0 for the reaction in 40% acetone–water. An interaction between the crystal violet ion and the carbinol occurs which tends to spurious rate constants if carbinol formation is slow ($t_{0{\cdot}5} > 1$ hr.). C. O. C.

Colour Reactions of the Ketohexoses. II— Blue Dye from Fructose and Diphenylamine Hydrochloride. H. Thies and G. Kallmich. *Chem. Ber.*, **85**, 438–443 (1952): *Chem. Abs.*, **46**, 9474 (25 Oct. 1952).

Ketohexoses and diphenylamine (I) in hot alcoholic HCl give a deep blue. Gently reflux sucrose (100 g.) in water (200 c.c.) and conc. HCl (1250 c.c.) with I (50 g.) in methyl alcohol (1250 c.c.) for 15 min., carefully add water (about 2500 c.c.) until the supernatant solution shows faint white opalescence caused by precipitation of unconsumed I, and filter off the foamy ppt., wash it free from acid, air-dry, and wash with petroleum ether to give 50–55 g. of a crude blue–black dye (II). II (50 g.) in $CHCl_3$ (250 c.c.) free from alcohol is passed through a 50 × 1·6 cm. acid-washed alumina column, and the chromatogram developed by passing chloroform (500 c.c.) containing 2% methyl alcohol, giving five zones— (1) blue–black, (2) deep blue, (3) olive green, (4) brown, and (5) light green. Zone 2 is eluted with chloroform–methyl alcohol (5 : 1, 250 c.c.) and the soln. poured into twice its vol. of petroleum ether, b.p. 120°c. After two to three additional precipitations from $CHCl_3$–CH_3OH into petroleum ether, 2 g. of II is obtained as a fine blue powder, which gives a uniform chromatogram on alumina. Shaking pure II (1 g.) in $CHCl_3$ (20 c.c.) with 5% KOH (20 c.c.) causes colour change from blue to red and then slowly to brown. Pouring the washed and dried $CHCl_3$ solution into petroleum ether causes separation of the dye base $(C_{15}H_{13}ON)_3$ (III) as a greenish-brown powder. Shaking the $CHCl_3$ soln. of III with HCl gives the blue dye salt again. III seems to be formed as follows—

$$2I + C_6H_{12}O_6 \rightarrow C_{36}H_{34}O_3N_2 + 4H_2O$$

probably via hydroxymethylfurfuraldehyde. III is not identical with the dye formed in the HNO_3 reaction (diphenyldiphenoquinonediimine), as shown by comparison of the ultraviolet absorption curves. Study of the absorption curve of III shows a certain similarity to that of asymmetrically substituted triphenylmethane dyes, which suggests that III has the structure—



although it could also have the structure—

$(C_6H_5)_2N{\cdot}C(CH_2OH){:}CH{\cdot}CH{:}C(OH){\cdot}CH{:}N(C_6H_5)_2]Cl$

by analogy with the polymethin dyes from furfuraldehyde and arylamines. C. O. C.

Hydroxyfuchsone Dyes. XVI— 2-Hydroxyfuchsone. I. S. Ioffe and B. G. Belenky. *J. Gen. Chem. U.S.S.R.*, **22**, 1437–1443 (Aug. 1952).

2-Hydroxyfuchsone is prepared by condensing resorcinol with diphenyldichloromethane, and its properties are compared with those of the isomeric benzaurin. In contrast with the latter, 2-hydroxyfuchsone is very unstable and forms salts only with acids. As in the case of unsubstituted fuchsone, these salts are stable only in strongly acid soln., and on partial neutralisation the colourless carbinol is formed. A. E. S.

Purpurogallin. X— Further Studies on the Oxidation Products of Purpurogallin and Purpurogallincarboxylic Acid. W. D. Crow, R. D. Haworth, and P. R. Jefferies. *J.C.S.*, 3705–3713 (Oct. 1952).

Absorption Spectra of Alkylazulenium Ions. L. H. Chopard-dit-Jean and E. Heilbronner. *Helv. Chim. Acta*, **35**, 2170–2193 (Dec. 1952).

The absorption spectra of azulene and 17 mono- and poly-alkyl deriv. in 50% sulphuric acid were determined in the range 220–400 mμ. The displacement of the maximum of longest wavelength was investigated, and a further example of a natural hypsochromic effect of an alkyl group discovered. Using the model proposed previously (*ibid.*, **35**, 1049 (May 1952)), semi-quant. molecular-orbital calculations were carried out, and found to explain the data obtained satisfactorily. H. E. N.

Azulenogen Content of Aqueous Camomile Preparations. II— Azulenogens in Infusions, Macerates, and Extracts prepared by Digestion. Z. Blažek and J. Hubík. *Czechoslov. Farm.*, **1**, 330–333 (1952): *Chem. Abs.*, **46**, 11,577 (25 Nov. 1952).

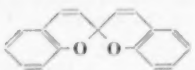
In infusion the azulenogen content diminishes as boiling proceeds. In macerates neither the temperature nor the time of extraction has any influence on the azulenogen content. C. O. C.

Some Adsorption Colours and their Significance for Thermochromic and Tautomeric Effects. III— Experiments with Anthrone Derivatives, Nitrotoluenes, *spiro*Pyrans, and Xanthhydrols. A. Schönberger, A. Mustafa, and W. Asker. *J. Amer. Chem. Soc.*, **74**, 5640–5643 (20 Nov. 1952).

Activated alumina added to a cold colourless soln. of 10-nitro(or anilino)anthrone turns deep orange, presumably because the coloured 10-nitro(or anilino)-anthranol is formed. Adsorptiochromism was also observed with the colourless 10:10′-dianthronyl and with 2:4-dinitrotoluene and 5-nitroacenaphthene. Cold or hot benzene soln. of dibenzo*spiro*pyran are colourless, but they produce deep violet on activated alumina or silica gel. Adsorptiochromism, piezochromism, and thermochromism are discussed in the case of some pyrans and pyrones and their sulphur analogues; replacement of O by S is detrimental to development of colour. C. O. C.

Formation of Coloured Forms of Spirans by Low-temperature Irradiation. E. Fischer and Y. Hirshberg. *J.C.S.*, 4522–4524 (Nov. 1952).

Colourless 1:3:3-trimethylindoline-2-*spiro*-6′-(2′:3′-β-naphthopyran) in ethanol–methanol solutions when irradiated for ~ 1 min. at below − 50°C. becomes intensely violet–red, while the colourless spiran (I) similarly affords an intense blue. This photoconversion does not occur with light of wavelengths > ~ 540 mμ. (i.e. energies lower than ~ 65 kg.cal./mole). It is held that, while configurational changes in the molecules are involved, bipolar mesomers do not contribute materially to the structure of the coloured modifications.



(I)

H. H. H.

Polymethin Dyes from 4:5-Disubstituted Thiazoles. E. B. Knott. *J.C.S.*, 4099–4106 (Nov. 1952).

α-Bromination and condensation with thioacetamide of α-aryloxy- and α-*p*-tolylthio-acetophenones give 4-aryl-5-aryloxy-2-methylthiazoles and 4-aryl-2-methyl-5-*p*-tolylthiothiazoles respectively, which are quaternised and converted into cyanines, carbocyanines, and dimethin-*mero*cyanines. Many of these dyes are good photographic sensitisers, but their most interesting property is their much higher solubility in organic solvents compared with closely related dyes from 4-arylthiazoles, benzothiazoles, and naphthothiazoles, a property held to be due to the fact that the aryl groups are non-planar. The weakening of the cohesive forces in the crystal resulting from a looser packing in order to accommodate the non-planar groups would then be expected to increase the solubility. The introduction of substituents into the 4-aryl groups, or the replacement of the 4-phenyl by the 4-β-naphthyl group, has little or no effect on the position of the absorption peak of any of these dyes. H. H. H.

Absorption Spectra of Thiacarbocyanine Dyes in Aqueous Gelatin Solutions. A. V. Borin. *Doklady Akad. Nauk S.S.S.R.*, **86**, 969–972 (11 Oct.). **Absorption Spectra of Thiacarbocyanine Dyes at the Interface between Silver Bromide and Gelatin Gel.** Idem. *Ibid.*, **86**, 1141–1143 (21 Oct. 1952).

An examination is made of the effects of various factors on the intensities of the *J*-bands (longer wave, attributed to an aggregated so-called "meso" form of the dye) and β-bands (shorter wave, attributed to the dimeric form of the dye) that often appear in the absorption spectra of solutions of thiacyanine dyes when gelatin is added. The dye examined is 3:3′-dimethyl-β-ethylthiacarbocyanine. It is shown that the β-band, but not the *J*-band, follows Beer's law as regards the effect of changes in concn. The intensity of the *J*-band is diminished and that of the β-band increased by (*a*) increasing the temp. of the gelatin soln. when preparing the mixture by adding molecularly dispersed dye (alcoholic soln.) to aq. gelatin, (*b*) decreasing the temp. of the dye soln. when preparing the mixture by adding an *aqueous* soln. of dye to aq. gelatin, and (*c*) vigorous agitation of the soln. during mixing— although subsequent agitation or heating is without effect. It is further observed that different qualities of gelatin give widely varying values for the relative intensities of the β- and *J*-bands, and, in order to determine the source of this variation, a gelatin giving predominant β-absorption is fractionated by treatment with water at 30–32°C. The more sol. fraction behaves very much like the original gelatin, but the less sol. fraction gives predominant *J*-absorption. Also, a gelatin giving predominant β-absorption is boiled in aq. soln. for some time, cooled to 40°C., and mixed with dye; predominant β-absorption is the result. When boiling is insufficiently prolonged (< 5 hr.), then if the gelatin is allowed to gel and left for 24 hr. before warming and mixing with dye there is a partial reversion in the character of the gelatin. The results indicate that the intensities of the *J*- and β-bands are affected in opposite senses by any given treatment, that the meso form of the dye is formed from molecularly dissolved dye, and that treatments calculated to increase temporarily or permanently the number of free side-chains in the protein lead to increase in β- at the expense of *J*-absorption. It is considered that the β-band is associated with the binding of dimeric dye molecules by the free side-chains, while the *J*-band is associated with the adsorption of monomeric or dimeric dye on the surface of the gelatin molecule, whereby the molecules take up positions parallel to the polypeptide chain and form into meso aggregates. Further experiments are carried out with films of fractionated gelatin formed on the surface of AgBr or of AgBr containing various amounts of Ag_2S; this surface is prepared by fusing the AgBr (+ Ag_2S) in a crucible and cooling. The surfaces of the film are stained with the dye and thin sections cut for examination. At the surface formed in contact with AgBr, β-absorption is increased and *J*-absorption diminished, these effects being greatly reduced by addition of Ag_2S to the AgBr. These effects are attributed to the orientation of polypeptide side-chains at an AgBr surface. A similar effect is found at an Hg surface. A. E. S.

***mero*Cyanine Dyes derived from Rhodanine. IV— Structure of the Decomposition Products of the Quaternary Salts of Dimethin*mero*cyanines.** Z. P. Sytnik, I. I. Levkoev, M. V. Deychmeyster, and L. D. Zhilina. *J. Gen. Chem. U.S.S.R.*, **22**, 1228–1234 (July 1952).

The authors have reported (see J.S.D.C., **67**, 318 (Aug. 1951)) that the unstable quaternary salts of rhodanine *mero*cyanines decompose in soln. to give a *mero*cyanine derived from thiazolidine-2:4-dione. However, van Dormael (cf. J.S.D.C., **66**, 155 (Feb. 1950)), from the elementary analysis of the decomposition product, considered it to be an α-mercaptoamide formed by rupture of the rhodanine ring, although, according to its reported physical properties, his substance was identical with the product obtained by Sytnik *et al.* Supplementary experiments confirm these authors' views, but it is shown that further treatment of the decomposition product with alcoholic potash leads to breakdown of the type postulated by van Dormael. The mercaptan itself, however, could not be isolated, as it is very readily oxidised to a disulphide. A. E. S.

Maurice Prud'homme. E. Justin-Mueller. *Teintex*, **17**, 723–727 (Nov. 1952).

A review of the contributions of Prud'homme to the colour-using industry, including the production of aniline black by the ferrocyanide process. B. K.

Sulphur Dyes produced by Sulphuration of Toluene-2:4-diamine. H. Hiyama, M. Ando, and T. Yamamoto. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **53**, 309–311 (1950): *Chem. Abs.*, **46**, 11,689 (25 Nov. 1952).

A brownish-yellow dye containing several 5(or 7)-aminobenzothiazole residues is obtained by fusing toluene-2:4-diamine with S at 250°C. C. O. C.

Synthesis of Organic Fluorescent Compounds. Z. Yoshida, Y. Shimada, and R. Oda. *Bull. Inst. Chem. Research, Kyoto Univ.*, **28**, 75 (1952): *Chem. Abs.*, **46**, 11,184 (25 Nov. 1952).

1*H*-Naphth[1:2]iminazole (yield 58·6%) and the 2-CH_3 (66%) and 2-C_6H_5 (53·4%) derivatives were synthesised by Fisher's method (*Ber.*, **34**, 934 (1901)). 2:2′-(*p*-Phenylene)- (40·2%) and 2:2′-(1:5-naphthylene)-bis(1*H*-naphth[1:2]iminazole) (61·4%) were synthesised by partial acylation in alcohol of 1:2-naphthylenediamine with the corresponding acid chlorides (the 1-NH_2 group was attacked), refluxing for 3–5 hr. in 4N-HCl or C_6H_5Cl, hydrolysis of the hydrochlorides obtained with aqueous alcoholic NaOH, and addition of and washing with water. Perimidine (47%) and its 2-CH_3 and 2-C_6H_5 derivatives (50%) were synthesised by Sachs' method (*Chem. Abs.*, **3**, 1870, 1982 (1909); **4**, 230 (1910)). 2:2′-(*p*-Phenylene)- (28·5%) and 2:2′-(1:5-naphthylene)-diperimidine (48%) were synthesised by adding small excess of the acid chlorides to 1:8-$C_{10}H_6(NH_2)_2$ in benzene while stirring at room temp., stirring for 15 min. more at room temp. and then for 15 min. at 60°C., dissolving the resulting hydrochlorides in a large quantity of hot water, neutralising with 10% ammonia, and washing with water. Perimidines are formed more readily than naphth[1:2]iminazoles, for the NH_2 groups in the 1:8 positions in the naphthalene ring are more reactive than those in the 1:2 positions. C. O. C.

Relation between Fluorescence and Chemical Constitution of Organic Compounds. Z. Yoshida, Y. Shimada, and R. Oda. *Bull. Inst. Chem. Research, Kyoto Univ.*, **28**, 76 (1952): *Chem. Abs.*, **46**, 11,185 (25 Nov. 1952).

When naphth[1:2]iminazoles (I) and perimidines (II) were irradiated with ultraviolet radiation (3600 A.), I fluoresced more than II. Introduction of a conjugated system in the 2-position of I increased fluorescence to some extent, but in the 2-position of II had little effect. I has strong blue–violet to greenish-blue fluorescence and is a useful fluorescent brightening agent. C. O. C.

Fluorescent Brightening Agents as Corrosion Catalysts. K. J. Nieuwenhuis. *Wasindustrie*, **1**, (5), 12–13 (1951): *Chem. Abs.*, **46**, 11,719 (25 Nov. 1952).

Fluorescent brightening agents act as corrosion catalysts in copper and brass washing machines where the work is washed at 60 and 80°C., rinsed, bleached at 25°C. with 0·25 g. active Cl per litre, treated with thiosulphate, and rinsed again. Combinations of fluorescent brightening agents added to one of the washes or rinses, chlorine bleach, reducing substances, and light seem especially harmful. C. O. C.

Pigments and Dyes for Camouflage and Infrared Photography. M. Déribéré. *Chim. Peintures*, **15**, 120–124 (1952): *Chem. Abs.*, **46**, 11,705 (25 Nov. 1952).

Recipes are given, the dyes being listed according to their reflective properties at 7800–8500 A. C. O. C.

Terminology of Artificial Colouring Materials. A. Lejeune. *Ing. Chim.*, **33**, 155–157 (1951): *Chem. Abs.*, **46**, 10,708 (25 Nov. 1952).

Colouring matters are given different names according to their use. A list of the various names applied to some common colours is given. C. O. C.

Colouring Matters of Flowers and Fruits— Anthocyanins and Flavones. C. Sannié and H. Sauvain. *Mém. Muséum natl. Hist. nat.* (Paris), [N.S.], **2B**, 254 pp. (1952): *Chem. Abs.*, **46**, 9795 (25 Oct. 1952).

A review including distribution, isolation, biological function, chemical and physical properties, and pharmacological action of the pigments. 700 references. C. O. C.

Synthesis of 3-Substituted Chromones by Rearrangement of *o*-Acyloxyacetophenones. W. D. Ollis and D. Weight. *J.C.S.*, 3826–3830 (Oct. 1952).

Stability of the Anthocyanin Pigments in Concord Grape Juice— Protective Action of Tannin. L. V. L. Sastry and R. G. Tischer. *Food Technol.*, **6**, 264–268 (1952): *Chem. Abs.*, **46**, 11,489 (25 Nov. 1952).

The principal pigment present was oenidin 3-monoglucoside. Exposure of juices and of purified pigments to ultraviolet radiation indicated that tannin had an important protective action on the anthocyanin. C. O. C.

Isolation and Identification of Kaempferol and Quercetin from Strawberries (*Fragaria chiloensis*). B. L. Williams and S. H. Wender. *J. Amer. Chem. Soc.*, **74**, 5919–5921 (5 Dec. 1952).

Chemistry and Utilisation of Travancore Minor Forest Products. IV— *Odina wodier* Bark. P. V. Nair and T. V. Punnoose. *Bull. Central Research Inst., Univ. Travancore*, **A 1**, (1), 87–102 (1950): *Chem. Abs.*, **46**, 11,675 (25 Nov. 1952).

Air-dried *Odina wodier* bark contains 8% tannin. An aqueous extract of the bark can be used for dyeing cotton, the fastness to light, washing, rubbing, and perspiration of the dyed cotton increasing in the order— Odina-dyed alone < with $CuSO_4$ < with $CuSO_4$ and chrome. Mordanting with $FeSO_4$, $Al_2(SO_4)_3$, $SnCl_2$, limewater, and $FeSO_4$ with chromic acetate yielded grey, buff–brown, yellow–brown, yellow–brown, and khaki, respectively. Odina-dyed cotton can be topped with basic dyes or coupled with Fast Salts. C. O. C.

Activation of the Fluorescence of Chlorophyll Solutions. R. Livingston and S. Weil. *Nature*, **170**, 750–751 (1 Nov. 1952).

Claims that the fluorescence of phaophytin is as intense in dry as in wet toluene are supported, but the suggestion that fluorescence of a toluene soln. of the Mg–phthalocyanine complex is increased by adding water is not upheld. On mixing dry toluene with an ethereal soln. of the complex and removing the ether a colloidal soln. is formed, the optical density of which decreases with time, a blue pigment ultimately settling to the bottom and leaving a clear supernatant liquid. Even so, the contention that the activator molecule adds to the central Mg atom rather than to ring V of the chlorophyll molecule is upheld. The equilibrium constant for formation of the addition compound is determined from the adsorption coefficient and the fluorescence intensity, using quinoline as activator and chlorophyll *a* as the pigment. Stability constants of various metallic pigment–solvent addition compounds are listed. J. W. B.

Polarisation of the Fluorescence of Chlorophyll a. R. Stupp and H. Kuhn. *Helv. Chim. Acta*, **35**, 2469–2482 (Dec. 1952).

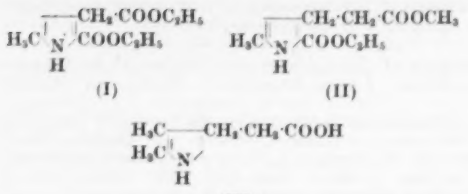
Labelling the peaks of the absorption spectrum of chlorophyll a— A, B, C, . . . starting with that of longest wavelength— Prins (*Nature*, **134**, 457 (1934)) supposed that the bands A–E were vibration bands belonging to the same electron transition. Solutions of chlorophyll a in castor oil and other solvents of high viscosity (which reduces the effects of Brownian movement) were irradiated with polarised, monochromatic light (λ = 360–660 mμ.), and the degree of polarisation p of the fluorescence measured —

$$p = \frac{I_{\parallel} - I_{\perp}}{I_{\parallel} + I_{\perp}}$$

($I_{\parallel}$ and $I_{\perp}$ = intensities of the fluorescence the electric vector of which is parallel and perpendicular respectively to the electric vector of the incident light). The fluorescence spectrum was independent of λ, but p was positive for λ = 360–520 and 560–660 and negative for λ = 520–560 mμ. It is shown therefore that Prins' hypothesis cannot be upheld. H. E. N.

General Route to ββ′-Substituted Pyrrole Intermediates for Porphyrin Synthesis. S. F. MacDonald. *J.C.S.*, 4176–4184 (Nov. 1952).

New pyrrole intermediates (I) and (II) are prepared by modifications of standard methods, the essential step being the substitution of benzyl for ethyl acetoacetate in the Knorr synthesis. A new synthesis of hæmopyrrole-carboxylic acid (III) is carried out via II.



(I) (II)

(III)

H. H. H.

Syntheses of Pyrroles, a Porphyrin, and the Maleinimide related to the Uroporphyrins. S. F. MacDonald. *J.C.S.*, 4184–4190 (Nov. 1952).

Representatives of the hitherto unknown pyrroles with both acetic and propionic acid residues in the β-position are synthesised (cf. *Chem. and Ind.*, 759, 1092 (1951)), including one of the hypothetical reduction products of the uroporphyrins. Also, a porphin-tetra-acetic-tetrapropionic acid is synthesised and oxidised to a carboxylated hæmatinic acid identical with that from natural uroporphyrin I. H. H. H.

Application of Fermentation Retting. Purification of Lac by Fermentation. H. Katagiri, N. Mugibayashi, and K. Morihara. *Bull. Inst. Chem. Res., Kyoto Univ.*, **18**, 41–43 (1949); **II.** H. Katagiri, N. Mugibayashi, and M. Omori. *Ibid.*, **20**, 67 (1950): *Chem. Abs.*, **46**, 10,637 (10 Nov. 1952).

I—Seed lac of low quality is purified by fermentation with *Pseudomonas vayzogenes* isolated from the seed lac. **II**—Total N after repeated alcohol extraction of seed lac was 3·9–5·8% on the dry weight. After 4 days' fermentation in Speakman's soln. at 35°c. the decomposition of dry matter, total N, hordein N, salt-sol. N, and glutelin N were 25, 23·5, 95·2, 50, and 17·7% respectively. Nearly 97% of the glutelin N was soluble in NaOH, and it contained 1·5% of chitin on its dry weight. C. O. C.

Studies in the Polyene Series. XLIV—Preparation of Octa-4-ene-2:7-dione and Octa-3:5-diene-2:7-dione, Intermediates for the Synthesis of Carotenoids. The Anionotropic Rearrangement of some Hexa-2:5-diene-1:4-diols. R. Ahmad, F. Sondheimer, B. C. L. Weedon, and R. J. Woods. *J.C.S.*, 4089–4098 (Nov. 1952).

Solubility of Melanins. A. J. Lea. *Nature*, **170**, 709 (25 Oct. 1952).

Substances are listed which dissolve, in the cold, pigments extracted from the ink sac of *Sepia officinalis* and melanoma tissue, and pigments prepared from adrenaline and 3:4-dihydroxyphenylalanine. The method of extracting the natural pigment is presumed to alter its chemical composition. J. W. B.

Pteridine Studies. III—Solubility and Stability to Hydrolysis of Pteridines. A. Albert, D. J. Brown, and G. Cheeseman. *J.C.S.*, 4219–4232 (Nov. 1952).

Wollastonite, a New Commercial Mineral. A. L. Hall, R. B. Ladoo, R. N. Secord, and C. A. Stokes. *Mining Eng.*, **4**, (10), 952–953 (1952): *Chem. Abs.*, **46**, 11,057 (25 Nov. 1952).

Wollastonite is essentially a Ca metasilicate, $CaSiO_3$. It is a white, medium hard, fibrous mineral with properties very similar to those of New York talc, which it may replace in paints. C. O. C.

A Pigment Particle Surface. E. J. Dunn. *Paint & Varnish Production*, **42**, (9), 19–24 (1952): *Chem. Abs.*, **46**, 10,638 (10 Nov. 1952).

Data are given to elucidate the surface composition of basic silicate white lead "45X". This pigment is prepared on the basis of an inert silica core with an active pigment coating. The different techniques used to demonstrate the presence and form of the sulphate-stabilised Pb silicate coating were—transmitted light microscopy, X-ray studies, chemical reactivity studies, electron microscopy, electron diffraction, settling tests, phase microscopy, and pigment extensions. Positive evidence is found of the presence of a coating of Pb compounds on the SiO_2 particles. The ultimate particle size in the Pb coating approaches colloidal dimensions, and may be one particle thick in some sections. When treated with K_2S soln., the "45X" particles show nearly completely black (PbS) discoloration; i.e. the surface of the SiO_2 particles is completely coated with Pb silicate and sulphate. C. O. C.

Pigments of Ural Sapropels. E. M. Titov. *Trudy Lab. Sapropel. Otlozheny Akad. Nauk S.S.S.R., Inst. Lesa*, (4), 29–39 (1950): *Chem. Abs.*, **46**, 10,307 (10 Nov. 1952).

The pigments in the sapropelite deposits of several Ural lakes are a mixture of carotenoids and fossil chlorophyll. The former consist mainly of β-carotene or at least of close stereoisomers. In calciferous this can reach 3 mg./100 g., while in siliceous forms it reaches 2 mg./100 g. The pink sapropels owe their colour to adsorbed carotenoids or mineral colloids, mainly $CaCO_3$. The chlorophyll arises from algæ, during the decomposition of which the chlorophyll is hydrolysed to chlorophyllide, which is stable for millenia, under anaerobic conditions in the lower layers. In Moltaevo Lake the sapropel layer on top of a peat layer contains no carotene but does contain a pigment not extractable by benzene though it is extracted by alcohol; it seems to be a derivative of chlorophyll b. C. O. C.

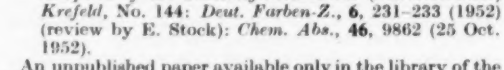
Manufacture of Alkali-resistant Prussian Blue. R. Beck. *Engineering Papers of the Dept. of Paints and Lacquers of the Textilingenieurschule (Färbereischule) Krefeld*, No. 144: *Deut. Farben-Z.*, **6**, 231–233 (1952) (review by E. Stock): *Chem. Abs.*, **46**, 9862 (25 Oct. 1952).

An unpublished paper available only in the library of the Textilingenieurschule. The stability of 200 mg. of Prussian blue in 10 ml. of 2% NaOH was determined as the time (in min.) it took to turn brown. Greatest alkali stability occurred when 9·5% Ni was present; it rose with increasing Ni content (from 1·4 to 9·5%). Alkali stability was lower in presence of Co and remained unchanged between 5 and 9% Co. Co combined with 1·4% Ni gave better alkali stability than 1·4% Ni alone. Increasing amounts of Ni and Co caused lowered tinting strength and a shift towards green. No truly alkali-stable Prussian blue has yet been made. C. O. C.

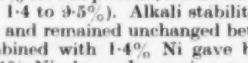
PATENTS

Intermediates for Cyanine Dyes. Ilford. *BP* 684,625

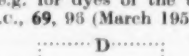
Intermediates, e.g. for dyes of the type described in *BP* 684,622 (J.S.D.C., **69**, 95 (March 1953)), of formula—



(R^1 and R^2 = Alk, hydroxyalkyl, or aralkyl; D = atoms to complete a 5- or 6-membered ring; n = 0 or 1) are obtained by reaction of 2 mol. of a compound of formula—



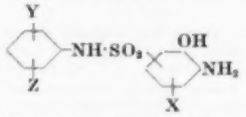
with > 1 mol. of a compound of formula $R^3{\cdot}NR^4{\cdot}CS{\cdot}S{\cdot}R^2$ (R^3 = Alk, aralkyl, or Ar; R^4 = H, Alk, aralkyl, or Ar) in presence of > 3 mol. of a compound of formula—



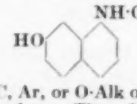
C. O. C.

Monoazo Metachrome Dyes. S. *BP* 683,891

Sulphonanilides of *o*-aminophenol—



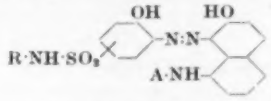
(X = H or Hal; Y and Z = H, Hal, Alk of 1–3 C; COOH, $SO_2{\cdot}NH_2$, $SO_2{\cdot}NH{\cdot}Alk$, or $SO_2{\cdot}NAlk_2$) are diazotised and coupled with a 1-acylamino- or 1-carbalkoxyamino-7-naphthol—



(R = Alk of 1–8 C, Ar, or O-Alk of 1–6 C) to give grey and blue metachrome dyes. Thus 2-aminophenol-4-sulphonanilide, diazotised and coupled in presence of soda ash with *N*-(7-hydroxy-1-naphthyl)adipamic acid, gives a grey. E. S.

Metal(Chromium)-complex Monoazo Dyes for Wool, Nylon, etc. Ciba. *BP* 684,646

oo'-Dihydroxymonoazo compounds containing a sulphamyl group but no sulpho or carboxyl groups—



(I)

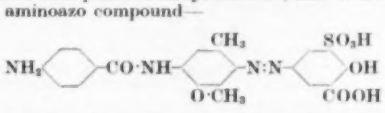
(R = H or Alk; A = acyl or carbalkoxy) are treated in substance with Cr compounds to give metal complexes containing < 1 Cr per mol. of monoazo compound. Mixed complexes are also made by chroming I in admixture with similar *oo'*-dihydroxymonoazo compounds derived from a 1-acylamino-7-naphthol containing neither sulpho, carboxyl, nor sulphamyl groups. The metal complexes so formed are more soluble in water than the unmetallised compounds, and dye wool, nylon, etc. from neutral or weakly acid baths. Thus, all parts being by weight, a mixture of the monoazo compounds 2-aminophenol-4-sulphonamide→1-acetylamino-7-naphthol (40) and 2-amino-4-chlorophenol→1-acetylamino-7-naphthol (355) suspended in water (3000) is boiled under reflux with a soln. (220) of sodium chromisalicylate of Cr content 2·6%. The product gives grey–blue on wool from a neutral or acetic acid bath. E. S.

Metallisable Direct Cotton Disazo Dyes. Ciba. *BP* 684,527

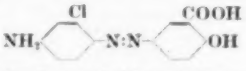
Dyes which may be metallised (especially with copper compounds) on the fibre or in the dyebath are made by combining together, by means of phosgene to form a urea derivative, 1 mol. of an aminoazo compound—

$R^1{\cdot}N{:}N{\cdot}R^2{\cdot}NH{\cdot}CO{\cdot}R^2{\cdot}NH_2$ or $R^1{\cdot}NH{\cdot}CO{\cdot}R^2{\cdot}N{:}N{\cdot}R^2{\cdot}NH_2$

with 1 mol. of an aminoazo compound $R^5{\cdot}N{:}N{\cdot}R^4{\cdot}NH_2$ (R^1 and R^5 = benzene series radicals containing OH *ortho* to COOH; R^2 = *p*-phenylene; R^3 = *m*- or *p*-phenylene; R^4 = benzene series radical in which the NH_2 is *para*, and a Cl atom is *ortho*, to the azo link. The finished dye molecule contains one SO_3H group, attached to R^1, or to either R^1 or R^5 if –N:N·R^2– is bound directly to R^1, and no COOH groups other than those in R^1 and R^5). Thus the monoazo compound 5-amino-3-sulphosalicylic acid→*p*-cresidine is condensed with *p*-nitrobenzoyl chloride, and then reduced to the aminoazo compound—



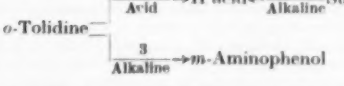
This is dissolved in aq. Na_2CO_3 together with 1 mol. of the aminoazo compound—



obtained by reduction of 2-chloro-4-nitroaniline→salicylic acid. Phosgene is passed in until amino groups can no longer be detected. The product dyes cotton yellow by the single-bath or two-bath aftercoppering process. E. S.

Black Trisazo Leather Dye. ICI. *BP* 684,352

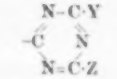
The trisazo dye—



gives level greys and blacks on leather, dyeing principally on the surface. It is less green in tone, and exhausts more completely from the dyebath, than the similar dye of *BP* 604,831 (J.S.D.C., **64**, 410 (1948)) derived from benzidine instead of *o*-tolidine. E. S.

4:4'-Diaminostilbene-2:2'-disulphonic Acids containing Cyanuric Rings— Fluorescent Brightening Agents. Ciba. *BP* 681,642

4:4'-Diaminostilbene-2:2'-disulphonic acids in which one H in each NH_2 is substituted by—



(Y = NH_2, NHR, or NR^1R^2; Z = Cl, O·Ar, S·R^3, or one of the groups described as Y) are fluorescent brightening agents with good affinity for vegetable and animal fibres. They are prepared e.g. by adding sodium 4:4'-diaminostilbene-2:2'-disulphonate to a suspension of cyanuric chloride in aqueous acetone and ice and then gradually introducing aq. Na_2CO_3. Phenol is next added, followed by aq. NaOH at 40–45°C., and finally monoethanolamine and aq. Na_2CO_3 at 75°C. R. K. F.

Halogenated Naphthaquinoneimine Derivatives— Dyes for Cellulose Acetate. S. *BP* 684,027

Halogenated naphthaquinoneimines which are blue dyes for cellulose acetate, fast to gas fumes, are prepared by (*a*) coupling 1 mol. of 1:5-dihydroxynaphthalene with 2 mol. of a diazo compound, halogenating the resulting disazo derivative, reducing to the diamine, and finally oxidising; or (*b*) halogenating 1:5-dihydroxynaphthalene, mono-nitrating or -nitrosating, reducing, and finally oxidising. The latter method gives a product with only one imino group. Thus, 1:5-dihydroxynaphthalene is coupled with diazobenzene, the product brominated in acetic acid at 80°C., and the resulting brominated disazo compound reduced with $SnCl_2$. The final oxidation is carried out by dissolving in NaOH and passing air through the solution. R. K. F.

Acylamino-2-anthraquinonyl Aryl Ketones— Vat Dyes. Ciba. *BP* 680,857

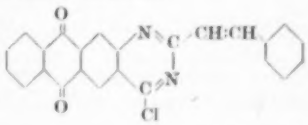
Grey vat dyes are prepared by acylating 2 mol. of an aryl 1:4-diamino-2-anthraquinonyl ketone (Friedel–Crafts reaction of the corresponding 2-carboxylic chloride with an aromatic hydrocarbon) with 1 mol. of an aromatic dicarboxylic dichloride. Thus fluoranthenedicarboxylic dichloride and 1:4-diamino-2-*p*-chlorobenzoylanthraquinone are heated at 160°C. for 3½ hr. in trichlorobenzene containing quinoline and pyridine. R. K. F.

1 - Acylamino - 2 : 4 - bis - β - hydroxyethylthio - anthraquinones— Dyes for Cellulose Acetate. Celanese Corpn. of America. *BP* 682,731

The 1-amino-2:4-bis-β-hydroxyethylthioanthraquinone of *BP* 678,644 (J.S.D.C., **68**, 526 (Dec. 1952)) is acylated with an aliphatic acid chloride or anhydride containing 2–6 C to give red dyes for cellulose acetate. R. K. F.

Quinazoline Derivatives. Ciba. *BP* 683,334

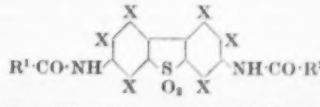
The quinazoline compound—



which gives green vat dyes when condensed with 4-aminoanthraquinone-2:1(*N*)-benzene-acridones, is prepared by first acylating 3-cyano-2-aminoanthraquinone with cinnamoyl chloride, and then cyclising the resulting product in nitrobenzene containing PCl_5 at about 180°C. R. K. F.

Fluorescent Brightening Agents. American Cyanamid Co. *BP* 684,842

Sulphonated derivatives of compounds of formula—



(X = H, Hal, Alk, OH, or O·Alk; R^1·CO and CO·R^2 = acyl radicals derived from aliphatic, araliphatic, alicyclic, aromatic, or heterocyclic carboxylic acids) have high affinity for cellulose, good fastness to light and washing, and strong bluish-white fluorescence. C. O. C.

Phthalocyanine Vat Dyes. Basf. *BP* 681,873

Metal-containing and metal-free phthalocyanines are treated with oleum or chlorosulphonic acid at 50–80°C. until soluble in alkaline aq. sodium hydrosulphite. The

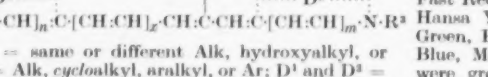
resulting products, containing one sulphonic acid group per 2–120 mol. of phthalocyanine, are blue vat dyes.
R. K. F.

Cobalt Phthalocyanine Derivatives. ICI. *BP* 681,917
Cobalt phthalocyanines are treated with a chloro- or bromo-methylating agent, e.g. *s*-dichlorodimethyl ether, in presence of $AlCl_3$ and a tertiary amine to give derivatives containing chloro(or bromo)methyl groups, useful for converting into the corresponding water-soluble phthalocyanines. Thus cobaltous phthalocyanine is heated at 50–55°C. for 30 min. with a mixture of $AlCl_3$, pyridine, and *s*-dichlorodimethyl ether.
R. K. F.

Water-soluble Cobalt Phthalocyanines. ICI.
BP 681,918
The cobalt chloro(or bromo)methyl-phthalocyanines of the previous patent are treated with a thiourea to produce water-soluble *iso*thiouronium derivatives useful for dyeing and printing. Thus cobaltous mono(chloromethyl)-phthalocyanine is heated at 100°C. for 10 min. with aq. *NNN'N'*-tetramethylthiourea, and the product separated after cooling by adding acetone.
R. K. F.

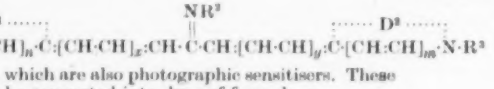
Phthalocyanine Vat Dyes. ICI. *BP* 681,929
Mixtures of cobaltous phthalocyanine and its chloro(or bromo)methyl derivative prepared as in *BP* 681,917 (above) are treated with a thiourea as in *BP* 681,918 (above) to give a product, containing 0·2–1 *iso*thiouronium group per mol. of phthalocyanine, which is a blue vat dye.
R. K. F.

Cyanine Dyes containing Amino Substituents in the Polymethin Chain. Ilford. *BP* 684,622
Dyes of formula—

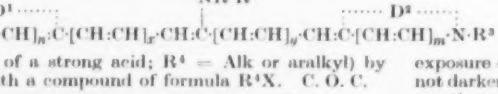


(R^1 and R^3 = same or different Alk, hydroxyalkyl, or aralkyl; R^2 = Alk, *cyclo*alkyl, aralkyl, or Ar; D^1 and D^2 = same or different atoms to complete a 5- or 6-membered ring; X = an acid radical; *m*, *n*, *x* = same or different 0 or 1) are useful as photographic sensitisers.
BP 684,650

Treating the above dyes with a strong base yields dyes of formula—

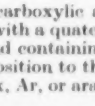


(y = 0 or 1), which are also photographic sensitisers. These in turn may be converted into dyes of formula—



(X = anion of a strong acid; R^4 = Alk or aralkyl) by treatment with a compound of formula R^4X.
C. O. C.

Dyes containing a Triazolone Nucleus. Ilford.
BP 684,707
Sensitising dyes are obtained by treating a compound of formula—



or the corresponding 4-carboxylic acid, in presence of a basic condensing agent, with a quaternary salt of a heterocyclic nitrogen compound containing a ·CH:CH·Q group in either the α or the γ position to the quaternary N (Q = SR^1 or NR^2R^3; R^1 = Alk, Ar, or aralkyl; R^2 and R^3 = H, acyl, or Ar).
C. O. C.

Recovery of Carbon Black from the Smoke produced in making Furnace Black. Phillips Petroleum Co.
BP 685,145
A method of forming an aqueous slurry of the carbon black contained in the smoke.
C. O. C.

Stabilising Calcium Lithol Toners. American Cyanamid Co. *USP* 2,601,468–9
Calcium lakes of lithol toners are rendered stable by heating them with mono-, di-, or tri-ethylene glycol, dioxan, or formamide, e.g. with diethylene glycol at 50°C. for 3–24 hr.
C. O. C.

Stabilised Barium and Strontium Lithol Toners. American Cyanamid Co. *USP* 2,601,668–9
Barium and strontium lithol toners are transformed physically so that they no longer change in colour when incorporated into vapour-setting inks and stored, and do not tend to cause the ink to body, by heating them with mono-, di-, or tri-ethylene glycol, dioxan, or formamide.
C. O. C.

Non-dusting Powders. YDC. *BP* 683,065
Powders, especially dye powders, are mixed with 0·1–2·0% on their weight of a liquid aliphatic alcohol containing >6 C. The resulting products are much less dusty while retaining their free-flowing character.
R. K. F.

Relation of the Fastness to Light of Organic Pigments to their Concentration in Mixtures with Substrates (V below).
Pearl Lustre and Essence— I (V below).
Metachromatic Effects of Anionic Polysaccharides and Detergents (VIII p. 102).

V— PAINTS; ENAMELS; INKS

Relation of the Fastness to Light of Organic Pigments to their Concentration in Mixtures with Substrates. R. Haug. *Farbe und Lack*, **58**, 340–344 (1952): *Chem. Abs.*, **46**, 11,704 (25 Nov. 1952).
Mixtures containing 20, 10, 5, 2·5, 1·25, 0·62, and 0·31% organic pigment and barytes were made by grinding barytes (80) with the pigment (20) and repeatedly diluting half the resulting "let-down" with an equal weight of barytes. The pigments used were Helio Fast Pink, Helio Fast Red RL, Lithol Rubine RK, Lake Red C (Ba lake), Hansa Yellow G, Permanent Yellow GR Extra, Helio Green, Pigment Green, Fanal Green, Fanal Blue, Water Blue, Madder Lake C. The pigment–barytes mixtures were ground in aqueous glue, painted on paper, and exposed to sunlight, their fastness to light being evaluated by plotting the results as trichromatic coefficients. The pigments generally progressed towards zero chroma along a straight line (their dominant wavelength). With Permanent Yellow GR and Lake Red C the chroma of 2·5% mixtures was higher than that of stronger mixtures; Lithol Rubine RK also showed this effect, but it was accompanied by a shift to purple. Water Blue faded more quickly, and Lithol Rubine RK, at higher strengths, darkened first; Lithol Rubine RK also darkened *in vacuo*, while its fading was suppressed (absence of water vapour). Characteristic curves were obtained for various pigments by plotting their relative brightness against pigment concn., and from these curves fading on light exposure could be calculated for those pigments which did not darken. Fading progressed essentially as a unimolecular reaction with $k = 7 \times 10^{-3}$ for Water Blue (poor fastness) and $k = 0{\cdot}2 \times 10^{-3}$ for Permanent Yellow (fairly fast).
C. O. C.

Pearl Lustre and Essence— I. J. Kishi. *Rept. Osaka Pref. Ind. Research Inst.*, **1**, 40–47 (1949): **II.** J. Kishi and S. Kato. *Ibid.*, **2**, 42–46 (1950): *Chem. Abs.*, **46**, 11,510 (25 Nov. 1952).
I— The abdominal pearly scales of *Trichiurus* sp. were freed from fat (15–20%) and water-soluble material (2–3%) and extracted with 0·2 N-NaOH to yield collagen (40–50%) and a filtrate, which was neutralised with acetic acid. The precipitate was filtered off, washed with acetone, and extracted with ethyl alcohol. The residue was separated with dil. ammonia to guanine (27–29%) and a purine-base glycoside (ca. 1%), and the extract further extracted with acetone to yield a crystalline lipoid B (0·7–0·9%), a sulphatide (a phosphatide with P replaced by S) which was decomposed to a fatty acid and oxidised to betaine. Direct extraction of the defatted scales with hot ethyl acetate gave, after cooling, lipoid A (1·8–1·9%), which was decomposed to a fat and a base. The scales were refined to a pearl lustre composed mainly of collagen and guanine by washing with water containing gelatin and casein, aq. (or gelatin) Na_2O_2, and/or ethyl acetate or ethyl phosphate. The refined scales (4%), mesityl oxide (or *cyclo*hexanone) (38), amyl acetate (28), butyl acetate (28), and celluloid (6) formed a good pearl essence.

II— Artificial pearl lustre was made by hydrolysis of $TiCl_3$ or $TiCl_4$ to $Ti(OH)Cl_3$, $Ti(OH)_2Cl_2$, and $Ti(OH)_3Cl$ (which is pearly iridescent), by dipping gelatin film into alcoholic $TiCl_3$ (12·2%) at 80° C. or alcoholic HCl (1·8)–TiO_3 [*sic*] (5·3%) at 50° C. Pearl lustre was also made by recrystallising $Pb(H_2PO_2)_2$ (from $Pb(NO_3)_2$ and $Ca(H_2PO_2)_2$) from alcohol. This showed pearly iridescence on films, and in amyl acetate or cellulose nitrate yielded a viscous liquid which could be applied to celluloid to produce imitation pearl effects. C. O. C.

PATENTS

Stable Aqueous Suspensions of Satin White. Albright & Wilson. *BP* 684,645

The satin white is intimately mixed with a protective colloid and the viscosity of the resulting mixture reduced by incorporating molecularly dehydrated alkali-metal or ammonium phosphate. C. O. C.

Polyvinyl Acetate Emulsion Paints. Shawinigan Products Corpn. *USP* 2,601,315

Modification of *USP* 2,567,678 (J.S.D.C., **68**, 224 (June 1952)), the process being much simplified and cheapened by eliminating use of heat. Incorporation of wetting agents produces a marked overall improvement in the performance of the paints. C. O. C.

VI— FIBRES; YARNS; FABRICS

Morphology and Chemical Composition of Certain Components of the Cotton Fibre Cell Wall. V. W. Tripp and M. L. Rollins. *Anal. Chem.*, **24**, 1721–1728 (Nov. 1952).

The work of the same authors and of M. S. Catlett *et al.* (J.S.D.C., **68**, 132 (April 1952)) is extended, furnishing a more detailed picture of the fine structure of the cotton fibre. The primary wall consists of a felted network of cellulose fibrils ~ 0·02 μ. in diameter and ~ 0·1 μ. thick, inclined at ~ 70° to the fibre axis, and embedded in a matrix of pectic, nitrogenous, and waxy substances. Its composition is ~ 50% cellulose, 9% pectic substances, 8% waxy material, plus nitrogen corresponding to 14% protein; there are traces of mineral matter and cutin. The secondary wall consists almost entirely of cellulose. The lumen is tubular in form, but shows no organised fine structure under the electron microscope; it contains desiccated protoplasmic remains rich in protein. Evidence is advanced to show that there is, between the primary and secondary walls, a "winding" of cellulose fibrils which is distinct from the regions bounding it, as has been noted by other workers. The winding is a layer, showing spiral structures at ~ 30° to the fibre axis, which is organised differently from both the primary and the secondary walls. Though not visible in unswollen fibres, it restricts swelling of the fibre by agents such as cuprammonium hydroxide. There are 7 photomicrographs and 4 electron-micrographs.

Abstractor's Note— Kling and Mahl (J.S.D.C., **68**, 224 (June 1952)), who have also noted the existence of an intermediate layer between primary and secondary walls, have since stated (J.S.D.C., **68**, 471–472 (Nov. 1952)) that there is no evidence that it is independent of the primary wall, but it is actually a part thereof. J. W. D.

Non-freezing Water and Non-freezing Benzene Capacities of Cottons and Modified Cottons. F. C. Magne and E. L. Skau. *Text. Research J.*, **22**, 748–756 (Nov. 1952).

The calorimetric heat of fusion method has been used to determine the amount of non-freezing water present in cotton and modified cotton fibres at various moisture contents and under various conditions. The non-freezing water capacity is apparently related to the total available surface, the proportion of amorphous cellulose, and the degree of distortion of the wetted fibres. A. B.

Mechanical Properties of Cellulose Fibres in relation to their Internal Structure. F. Andersen. *Contrib. Danish Inst. Textile Research* (in *Trans. Danish Acad. Tech. Sci.*), (3), 75 pp. (1950): *Chem. Abs.*, **46**, 9837 (25 Oct. 1952).

The static and dynamic (very low amplitude; 25–40 cycles/sec.) moduli of elasticity E of cotton and rayon fibres have been determined. During first stretch irreversible changes occur owing to intermolecular flow and improved alignment in the amorphous region. The fibre is now mechanically conditioned. Successive cycles are essentially reversible providing maximum tension is not > that in the first cycle. At every stretch, relaxation occurs, decreasing or increasing to the steady value on contraction. With cotton, stress–strain curves are linear at all R.H. (0–100%) up to the ultimate strength. With increasing R.H., stiffness decreases. With rayon, the curves are non-linear, complicated, and greatly dependent on R.H. When cotton is immersed in water, E is very small at first but increases with stretch, so that eventually it equals or exceeds the dry value. With cotton the dynamic E is independent of R.H. but increases with degree of stretch. With rayon, the dynamic E depends on R.H. but remains constant at different degrees of stretch. Increasing temperature decreases the static E for rayon and cotton and the dynamic E for rayon. Relaxation effects at constant length can be described by Eyring's theory based on the existence of a viscous unit. Relaxation is quicker the higher the moisture content, and smaller the greater the degree of stretch. Static E depends on structure and arrangement of the amorphous regions, while the dynamic E is determined by the crystalline region. In cotton arrangement of molecules is such that tension occurring during stretching is unevenly distributed among the molecules, whereas on rayon it is evenly distributed. Water facilitates internal flow. At high degrees of stretch, distribution of stretch in cotton in water becomes uniform and modulus increases rapidly. C. O. C.

Influence of Solutions of Salts on the Elastic Properties of Cellulose Fibres. R. W. Asmussen, F. Andersen, and B. Krawack. *Contrib. Danish Inst. Textile Research* (in *Trans. Danish Acad. Tech. Sci.*), (5), 13 pp. (1950): *Chem. Abs.*, **46**, 9839 (25 Oct. 1952).

With most salt solutions, concentration even at saturation is not very high, so that the fibre elasticity is determined mainly by the water molecules. Na^+, K^+, Br^-, and F^- have no effect, but > 20% aq. $ZnCl_2$ does have an effect in the case of rayon and > 61% $ZnCl_2$ in the case of cotton. With increasing concentration the modulus increases; with increasing time of contact with $ZnCl_2$ soln. the modulus decreases. C. O. C.

Degradation of Cellulosic Fibres in Acid Medium. P. Rodet. *Ann. Chim.*, [xii], **6**, 786–834 (1951): *Chem. Abs.*, **46**, 9839 (25 Oct. 1952).

A study of the effect of the fibre micellar structures in acid degradation. Cotton apparently consists of crystallites 600–650 A. long and 50–100 A. wide, joined together by regions in which the molecules form microfibrillary elements 2000–2500 A. long and 300 A. in diameter. The structure of mercerised cotton is very similar to that of native cotton, but the swelling caused by mercerisation leads to different crystalline orientation of the molecules and produces tensions and distortion within the fibre; these prevent recovery of initial crystallinity but permit some reorganisation of molecules during hydrolysis. The micellar structure of rayon could not be determined accurately by hydrolysis, as the swelling affected hydrolysis and recrystallisation. The length of the final crystallite seemed to depend on the experimental conditions without being affected by the type of micellar orientation of each fibre. The curve of variation of the degree of polymerisation characterised each fibre (all other factors being equal), and change in the shape of the curve indicated modification of the structure and change of physical properties. Little information was obtainable on the hydrolysis itself. It was verified, however, that the reaction is catalytic and that H_3O^+ ions act as catalyst, although their action was difficult to ascertain because of osmotic phenomena occurring on the fibre surface and of swelling, which enhanced penetration of reagents into the fibre. C. O. C.

Study of the Addition of Phenyl *iso*Cyanate to the Hydroxyl Group of Cellulose. P. Schneebeli. *C.R. Acad. Sci. Paris*, **234**, 738–740 (1952): *Chem. Abs.*, **46**, 10,613 (10 Nov. 1952).

Phenyl *iso*cyanate, dry pyridine, and dry cellulose were heated at 80 and 100° C. Curves show the degree of esterification as a function of time and temp.; there is an induction period. X-Ray examination revealed that at first esterification did not affect the crystalline regions of the fibre, but after > 21·4–36·6% of the OH groups had been esterified esterification began in the crystalline regions.

At a high degree of esterification the last traces of cellulose disappear. The amount of water adsorbed at 40 and 81·5% R.H. passes through a minimum when the degree of esterification is 25%. C. O. C.

Practical Application of the Iodine Value Conversion Diagram in Studies of Crystallisation Processes. K. Schwertassek and K. Hájek. *Faserforsch. und Textiltech.*, **3**, 349–354 (Sept. 1952).

The relationship previously found between crystallinity and iodine absorption value (see J.S.D.C., **68**, 541 (Dec. 1952)) is used to follow changes occurring in viscose rayon and cotton in the course of alkaline treatments. Treatment of viscose rayon (67% crystalline) in 6% aq. NaOH at 20°c. for 30 min. leads to dissolution of 4·9% of the cellulosic material. This, in itself, should cause the crystallinity of the residue to increase to 70·5%, whereas the figure found from iodine absorption measurements is 81%, thus indicating that a crystallisation process has occurred. Owing to the nature of the non-cellulose impurities, it is impossible to determine the crystallinity of raw cotton directly, but degreased, demineralised, kier-boiled cotton (practically 100% cellulose) contains 97% of crystalline cellulose. A. E. S.

Effect of Moisture on Compressibility of Natural High Polymers. C. E. Weir. *Bur. Stand. J. Res.*, **49**, 135–139 (Sept. 1952)— *Research Paper* 2349.

The general behaviour of leather, cellulose, wool, and silk fibroin is similar. For low moisture contents, compressibility is essentially independent of moisture content; for high moisture contents, compressibility increases markedly. It is suggested that these data are consistent with the concept that moisture exists in hydrogen-bonded form at low, and as liquid water at high, moisture contents. The range of moisture content within which compressibility becomes more marked corresponds to that in which the slopes of differential heat of adsorption curves change most rapidly, and in which electrical properties begin to change rapidly. Empirical compression equations are derived from the data—

For dry wool—

$$-\Delta V/V_0 = 1{\cdot}37 \times 10^{-5}(P - 2000) - 6{\cdot}79 \times 10^{-10}(P - 2000)^2 + 1{\cdot}55 \times 10^{-14}(P - 2000)^3$$

For silk fibroin—

$$-\Delta V/V_0 = 1{\cdot}24 \times 10^{-5}(P - 2000) - 7{\cdot}00 \times 10^{-10}(P - 2000)^2 + 2{\cdot}68 \times 10^{-14}(P - 2000)^3$$

(P is expressed in atmospheres). J. W. D.

Textile Boom in Fourteenth-century York. J. N. Bartlett. *J. Bradford Textile Soc.*, 57–60 (1951–52).

Historical. In the late fourteenth century clothworkers were scattered throughout the West Riding, but the specialised and dominant centre was York. C. O. C.

Story of Wool in the West Riding. R. Patterson. *J. Bradford Textile Soc.*, 46–51 (1951–52).

Historical. C. O. C.

William Greenwood and Robert Heaton—Two Eighteenth-century Worsted Manufacturers. E. M. Sigsworth. *J. Bradford Textile Soc.*, 61–72 (1951–52).

A brief account of the records of William Greenwood and Robert Heaton, who in the late eighteenth century were worsted manufacturers doing their own combing and dyeing, in Oxenhope and Haworth respectively. C. O. C.

Is α-Keratin a Closed Coil? F. H. C. Crick. *Nature*, **170**, 882–883 (22 Nov. 1952).

Occurrence in α-keratin of the 1·5 A. meridian reflection, characteristic of the Pauling–Corey α-helix structure for poly-γ-methyl-L-glutamate, suggests that the α-helix forms an important part of α-keratin. This can be reconciled with the 5·15 A. reflection, very close to the meridian, of α-keratin by bending the α-helix, which shows a 5·15 A. reflection displaced from the meridian, into a super-helix or coiled coil. Consideration is given to the necessary energy and spatial changes, and it is suggested that the α-helix can be deformed an appreciable amount if the deformation is systematic. J. W. B.

Mechanism of the Elongation of Hairs. W. Fuhrmann. *Melliand Textilber.*, **33**, 911–915 (Oct. 1952).

The characteristic load-elongation diagram of keratin fibres is interpreted in terms of a fibre model consisting of rubberlike spindle-shaped units embedded in a plastic medium. During the initial rapid rise in load with elongation, it is essentially the spindles that take the load, but during the slow rise in the middle portion of the curve a critical shearing stress is progressively attained in the interstitial medium at various points along the spindles, and relative movement of the spindles occurs. When the yield point of the medium has been attained at all points, a condition of rapid rise of load with elongation again sets in. Various mechanical and chemical treatments of the fibre may affect the yield point of the interstitial material and the tension-dependent and tension-independent components of the elastic modulus of the spindles, and the effects of such changes are predicted from a theoretical consideration of the model. Load–elongation curves are determined for a hair subjected to repeated extension–relaxation cycles and for hairs treated in solutions of phenol, of thioglycollates, of hydrogen peroxide, and of formaldehyde; they are interpreted on the basis of the model. A. E. S.

Displacement of the Iso-ionic Point of Proteins during Deformation. A. Pasynsky and V. Blokhina. *Doklady Akad. Nauk S.S.S.R.*, **86**, 1171–1173 (21 Oct. 1952).

Unloaded wool, wool under tension (30% extension), and supercontracted wool (20% supercontraction) are each immersed in water and titrated potentiometrically with HCl in order to determine the iso-ionic point. The values obtained are pH 5·5, 5·29, and 5·8 respectively. References are made to similar observations on muscle protein. It is considered that increased extension leads to the freeing of some of the COO^- and NH_3^+ groups from their association with polar groups, and that the difference in the energies of the two types of hydrogen bond so broken (–COO⁻···HO– and $\overset{+}{\text{N}}$H···HO–) accounts for the displacement in pH that occurs at the iso-ionic point. The displacement observed (± 0·3) corresponds to the blocking or freeing of about 15% of the total number of carboxyl groups present in the keratin. A. E. S.

Chemical Mechanism of Permanent Set. R. S. Asquith and J. B. Speakman. *Nature*, **170**, 798–799 (8 Nov. 1952).

The concept of chemical mechanism in permanent set in strained wool fibres is upheld by experiments in which the ε-*N*-dinitrophenyllysine content of fibres, set in boiling borax soln., treated with 1-fluoro-2:4-dinitrobenzene, and hydrolysed, is measured after separation on a silica column. Lysine content ranges from 2·15 to 2·53% compared with 3·27% for untreated wool and 3·33% for set fibres hydrolysed without prior fluorodinitrobenzene treatment. Clearly, lysine side-chains take part in the new cross-link formation, the links being broken by acid hydrolysis. This is compatible with the formation of CH:N or S·NH bonds in set fibres. J. W. B.

Kinetics of Anion Exchange on Wool. R. F. Hudson and G. A. Schmeidler. *J. Phys. Colloid Chem.*, **55**, 1120–1130 (1951): *Chem. Abs.*, **46**, 11,692 (25 Nov. 1952).

Study of the kinetics of exchange of bisulphite ion by other anions showed that the calculated diffusion coefficients decrease and activation energies increase in the order NO_3^-, Cl^-, SO_4^{2-}, $C_6H_5{\cdot}SO_3^-$, $H_2PO_4^-$, which thus represents the ease of diffusion of these ions into the fibre. Systematic change in diffusion coefficient with viscosity of the salt solution suggests that migration proceeds through the aqueous phase within the fibre, although the activation energy of 10–14 kg.cal./mole is much greater than that for diffusion in aqueous soln. This high value is attributed to the gel structure and low mobility of absorbed water in the micropores of the fibre. Regeneration experiments show that the rate depends on the penetrating basic ion. The rate of desorption of bisulphite ion from wool in alkaline soln. is much greater than that at neutrality; at pH 7·4 $D_f = 0{\cdot}77$, and at pH 13 $D_f = 3{\cdot}24$ sq.cm./sec., both at 18°c. C. O. C.

Action of Nitrous Acid on Wool. F. Hirsch. *Melliand Textilber.*, **33**, 941–943 (Oct. 1952).

Load–extension curves are obtained for keratin fibres by subjecting them to successive small const. increases in load and allowing equilibrium elongation to be attained at each stage. They have the characteristic three approximately linear sections, and, for the transition points between the first and middle sections (I) and between the middle and

final sections (2), and for the breaking point (3), a function $R = 100lF_0f/l_0P$ may be written (l_0 = unloaded length, l = loaded length, F_0 = initial cross-sectional area, P = load), in which the factor f has such values (f_1, f_2, and f_3) at the points (1), (2), and (3) that for the *untreated* fibre R has the same value at each of these points ($R_1 = R_2 = R_3 = R_0$, the ideal value). The values of f_1, f_2, and f_3 depend on the type of hair or wool. For *treated* fibres, we may write a damage factor, $D = R_1 + R_2 + R_3 - 3R_0$, which is considered to give a measure of chemical damage. The method is applied to wool, both unreduced and reduced with thioglycollic acid, after treatment with nitrous acid. From the results obtained and from other observations it is concluded that nitrous acid reacts not only with amino groups, but also with sulphur-containing groups. In particular, nitrous acid reacts with the cysteine residue, giving a deriv. that is unstable to alkali; this may react further with rebuilding of disulphide cross-links. A. E. S.

Behaviour of Protein Fibres and Polyamides toward Acids and Bases. I— Absorption of Hydrochloric Acid. H. G. Fröhlich. *Deut. Textilgewerbe*, **52**, 397–398 (1950): *Chem. Zentr.*, **II**, 1762–1763 (1950): *Chem. Abs.*, **46**, 11,691 (25 Nov. 1952).

The curves for absorption of HCl by Ardil, zein, and casein fibres have the same shape as those for silk and wool. At pH 1·3–0·8 there is a region of constant acid fixation. Below pH 0·8 rise in absorption occurs, its steepness varying with the different fibres. H·CHO determinations showed that the varying rates of increase in acid fixation cannot be explained by differences in intensity of the splitting off of H·CHO. Rather, they are concerned with addition of protons to the negatively charged O of the peptide group. The slight absorption of HCl at pH < 0·8 is due to the ability of the polypeptide groups to attract and bind protons with salt formation, which is reduced in proportion to the H·CHO content. The absorption curves for nylon and Perlon U are also similar to those of wool and silk, absorption increasing with decreasing pH and, with nylon, reaching saturation at pH 2·2–2·6. C. O. C.

Relations between Small-angle Interferences of Silk and the Wax Content. O. Kratky, E. Schauenstein, and A. Sekora. *Nature*, **170**, 796–797 (8 Nov. 1952).

Production of new equatorial small-angle reflections on treating fresh preparations of silk fibroin with alkali is now supposed to be due to waxy substances. The reflections disappear after solvent extraction of the material, 1·6% (on wt. of degummed silk) of wax being removed. The extracted wax shows sharp reflections corresponding to net-plane distances which are in good agreement with those of alkali-treated fresh silk. Ultraviolet absorption bands characteristic of conjugated unsaturated fatty acids are also found. It is suggested that hot alkali brings the primarily amorphous waxy substances to crystallisation, a process more easily effected with fresh samples which have well formed intermicellar spaces than with older preparations with probably closer packed units. There appears to be some interaction with the polar side-chains of the fibroin. A functional significance for the silk fat in the transformation of the globular into the fibrous form is tentatively suggested. J. W. B.

Kinetic and Thermal Study of the Polymerisation of Caprolactam. S. M. Skuratov, A. A. Strepikheev, V. V. Voevodsky, and E. N. Kanarskaya. *Doklady Akad. Nauk S.S.S.R.*, **86**, 1155–1158 (21 Oct. 1952).

The course of the polymerisation of ε-caprolactam at 231·5°C. in presence of varying amounts of water, and at various temp. (200–240°C.) in presence of a const. amount of water (1·96%), is followed by means of a delicate calorimetric method of measuring the rate of heat evolution (the heat of reaction is found to be 3·26 kg.cal./mole). Water is essential for the reaction, and, as the water content increases, the period of induction diminishes and the reaction rate increases. It is considered that the first stage in the reaction is the hydrolysis of a very small amount of lactam to amino acid, which reacts with more lactam to give dimer, which reacts with lactam to give trimer, and so on; a kinetic equation is worked out on this basis. In agreement with experimental results, the equation predicts that the max. reaction rate will be directly proportional to the water content at a given temp. and will occur at 42% conversion into polymer for all values of temp. and water content. A. E. S.

Important Physical Properties of Perlon Fibre. H. Böhringer. *Faserforsch. und Textiltech.*, **3**, 381–390 (Oct. 1952).

A comprehensive review is made of the relations existing between the various physical properties of Perlon filaments and yarns, including degree of stretch, yarn-construction parameters, and the elastic and tensile properties. Also, the effects of temp. and R.H. on these properties are considered. There are 26 graphs. A. E. S.

Dihalogeno-substituted Terephthalaldehydes and *p*-Tolualdehydes. J. R. Naylor. *J.C.S.*, 4085–4086 (Oct. 1952).

2:5-Dichloro- and 2:5-dibromo-terephthalaldehydes are conveniently prepared, but not in good yield, by oxidation of the corresponding dihalogeno-substituted *p*-xylene with chromium trioxide and sulphuric acid in the presence of acetic acid and acetic anhydride, to the corresponding terephthalaldehyde tetra-acetates, from which the free aldehydes are obtained in almost quantitative yield by hydrolysis with dil. H_2SO_4. The new 2:5-dichloro- and 2:5-dibromo-4-methylbenzaldehyde diacetates are obtained as by-products of the oxidation, and are readily hydrolysed to the aldehydes. H. H. H.

PATENTS

High-tenacity Viscose Rayon. Comptoir des Textiles Artificiels. *BP* 685,631

Viscose rayon of dry tenacity > 5 g./denier, wet tenacity 3 g./denier, and elongation > 7% is produced by extruding into a coagulating bath containing 10–15 g. acid per litre at 15–30°C. a viscose of viscosity > 400 poises and a γ-value > 60, and then setting the filaments in hot water. W. G. C.

Viscose Rayon Staple. Glanzstoff-Courtaulds. *BP* 685,460

Viscose staple of improved quality is obtained by delaying the cutting process until the chemical and washing aftertreatment. W. G. C.

Regenerated Protein Fibre. Courtaulds. *BP* 684,506

Fibres are made by extruding a solution of protein in di- and/or tri-chloroacetic acid into a non-solvent coagulating bath which is miscible with the above acids, e.g. water. W. G. C.

Stabilising Polythene to Sunlight. ICI *BP* 684,976

Protection on exposure to bright sunlight is given by incorporating a small amount of an Ni salt of an acid of formula $R^1{\cdot}NR^2{\cdot}CSSH$ (R^1 and R^2 = alkyl radicals together containing < 8 C). C. O. C.

Polyvinyl Chloride Fibres. Société Rhodiacéta. *BP* 685,077

The spinning liquid is obtained by treating the polymer with a non-solvent liquid swelling agent, of which the polymer can absorb at least its own weight to yield a homogeneous product from which the swelling agent cannot be decanted. C. O. C.

Rapid Ageing of Polyamides containing Hydroxyl Groups. Organico. *BP* 684,972

Polyamides containing hydroxyl groups are rapidly aged by treatment at > 50°C.; e.g. 90 hr. at 158°C. gives the same effect as storing at room temperature for 3 months. C. O. C.

Polyurethans. Celanese Corpn. of America. *BP* 685,729

Polyurethans suitable for spinning into fibres are obtained in good yield by mixing a diamine, e.g. tetramethylenediamine, and a bischloroformate, e.g. tetramethylene bischloroformate, so that they do not react together, and then bringing about reaction by adding an alkaline catalyst, e.g. sodium carbonate. W. G. C.

Solvent for Polyacrylonitrile. DuP. *BP* 685,386

Acrylonitrile polymers containing > 85% acrylonitrile are soluble in *NN*-dimethylacetamide to give solutions which are less discoloured than dimethylformamide solutions and are suitable for spinning into fibres, etc. W. G. C.

Acrylonitrile Polymers. Industrial Rayon Corpn. *USP* 2,601,251

Copolymers of acrylonitrile and a monovinyl ether of an aminoalcohol can be formed into fibres of high tensile strength which are easily dyed with acid dyes.

USP 2,601,252

Copolymers containing $> 85\%$ acrylonitrile may be dissolved in a mixture of nitromethane (9·0–9·8 parts) and formamide (1).

USP 2,601,253

Discoloration in spinning solutions of copolymers containing $< 80\%$ acrylonitrile is inhibited by incorporation of a small amount of a water-soluble, hydroxy-aliphatic mercaptan containing only one free –SH group.

USP 2,601,254

A mixture of ethylene carbonate and a water-soluble polyhydric aliphatic alcohol of formula—

$$HO{\cdot}[CH_2{\cdot}CHR{\cdot}O]_n{\cdot}H$$

(R = H, CH_3, or CH_2OH; n = 1–4 when R = H, 1–3 when R = CH_3, and 1 when R = CH_2OH) is a solvent for copolymers containing $< 80\%$ acrylonitrile.

USP 2,601,255

Discoloration of cyclic carbonate spinning solutions of acrylonitrile polymers is inhibited by incorporation of a colourless compound containing two ketonic carbonyl groups, with only one H-bearing C atom between them, the remainder of the compound consisting of C and H, e.g. acetylacetone.

USP 2,601,256

Copolymers of acrylonitrile and compounds of formula $CH_2{:}CR{\cdot}CH_2{\cdot}[A]_n{\cdot}SO_3H$ (n = 0 or 1; R = H or CH_3; A = oxyalkylene, hydroxyalkyleneoxy, thioalkylene-hydroxy, or thioalkylene of > 1 C) are suitable for forming into orientable fibres of improved hydrophilic properties.

C. O. C.

Fibre-forming Linear Polyhydrazides. Celanese Corpn. of America. *BP* 684,699

Linear thermoplastic fibre-forming polyhydrazides are obtained by reaction of a dicarboxylic acid, ester, halide, or anhydride with a dihydrazide of a dicarboxylic acid.

C. O. C.

Stabilising Threads of Polydiene–Sulphur Dioxide Compounds. Courtaulds. *BP* 684,967

Aromatic hydroxy compounds, e.g. pyrogallol, catechol, or β-naphthol, stabilise polydiene–sulphur dioxide compounds to atmospheric conditions, heat, and light.

C. O. C.

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Wetting Power from the Physicochemical Point of View. G. Knafo, G. Reutenauer, and L. Rosano. *Teintex*, **17**, 786–797 (Dec. 1952).

Measurements of surface tension and its variation with time, adhesion tension for paraffin wax, penetration of solution into wool felt, and wetting power have been made using solutions of eight commercial wetting agents at concentrations of 0·005–4·0 g./litre. Adhesion tension is defined as $\gamma \cos \theta$ (γ = surface tension; θ = angle of contact). Results suggest that, where wetting occurs without penetration, the adhesion tension is the important factor. Where penetration into material occurs, the concentration of the wetting agent is also important, and it seems that considerable imbibition takes place only when the concentration at which micelle formation occurs is exceeded. W. R. M.

New Problems encountered by the Garment Dyer and Cleaner. M. Marcou. *Teintex*, **17**, 629–641 (Oct. 1952).

Older types of finishing agents such as starches are easy to remove and do not interfere with subsequent dyeing or dry cleaning. This is not the case, however, with the newer types of finishes, which include crease-resist, non-shrink, embossed, and chintz finishes and also water-proofing treatments. Methods of processing garments containing these finishes are described. B. K.

Resistance of Bacteria to Quaternary Compounds. R. Fisher and P. Larose. *Nature*, **170**, 715–716 (25 Oct. 1952).

Bacterial cultures are developed under conditions which lead to an increased resistance to the cationic detergent alkylbenzyldimethylammonium chloride. Adaptation is dependent on the pH of the medium, and the relation between the bactericidal activity of a compound and its affinity for wool is emphasised. J. W. B.

Degreasing Woolskins. R. F. Innes and K. G. A. Pankhurst. *J. Soc. Leather Trades Chem.*, **36**, 358–363 (Nov. 1952).

Woolskins are degreased by paraffin degreasing processes normally applied to sheep pelts, to establish whether undue felting takes place. Serious trouble is not encountered up to 2 hr. 20 min. drumming in paraffin at 12 r.p.m. Good results are obtained when the skins are dry-pickled and then painted with a mixture of equal volumes of an alkylphenol–ethylene oxide condensate (Corilene DG) and paraffin, piled flesh to flesh overnight, and rinsed in the morning in a paddle of warm water.

J. W. B.

Laundering of Wool—Effect of Detergents and Washing Methods on Fabric Properties. M. S. Furry and E. M. O'Brien. *Amer. Dyestuff. Rep.*, **41**, 763–769 (24 Nov. 1952).

On repeated (10 times) laundering of a light-weight wool fabric, dyed with acid dyes, in a Launderometer, using various detergents and washing conditions, it was found that shrinkage and change in breaking strength and elongation were related to the severity of agitation rather than to the temperature of the wash. The different detergents only caused differences in shrinkage when agitation was severe, more shrinkage being caused by built synthetics than by unbuilt synthetics or soap. Repeated laundering caused slight loss of strength and increase in elongation, but the difference between detergents was only slight. Loss of colour and chemical damage (as measured by indigo carmine absorption) were chiefly influenced by the temperature and duration of the wash and the pH of the detergent solution; the degree of agitation had little effect. Thus a long soak wash may be more injurious to wool than a short wash with mild agitation. W. K. R.

Effect of Dry Cleaning Solvents on Manufactured Fibres. [J. Pinte.] *Teintex*, **17**, 497–501 (Aug. 1952).

Viscose and acetate rayons, nylon, and vinyl chloride were subjected to the action of dry cleaning solvents (trichloroethylene, perchloroethylene, benzine, petrol, solvent naphtha, and white spirit) under conditions corresponding to bulk operation. Tensile strength, resistance to stretch, swelling, and degree of polymerisation tests showed that viscose and acetate rayons and nylon were unaffected, whilst vinyl chloride was affected by benzine and trichloroethylene. B. K.

Accelerated Boiling of Open-width Cotton Cloth. V. E. Rostovstsev. *Ivanov. Nauch.-issledovatel. Inst. Khlopchatobumazh. prom., Nauch-issledovatel. Trudy*, **18**, 76–89 (1951): *Chem. Abs.*, **46**, 9853 (25 Oct. 1952).

An investigation of the accelerated treatment of cotton cloth with hot aq. soln. Various forms of apparatus were used including multiple baths with submerged squeeze rollers, feeding the cloth by submerged rollers past a vacuum nozzle, and forcing the liquor under pressure through the cloth as it passes through the bath. The results obtained by alkaline boiling indicate that appearance of capillary properties in boiled cloth is caused not only by removal of waxes but also by some destruction of the primary cuticle of the fibres. The multiple squeezing method at 95°C. is effective in accelerating the process only if there are at least 40 squeezes. The vacuum method is very effective with linear velocities of liquor of 0·4–6·5 m./sec., giving good capillarity of the cloth with a total pass-through time of 0·2–0·25 sec. per unit area of cloth. Very good acceleration is achieved by the pressure impregnation method for 0·36–0·45 sec. at 95°C. At linear speeds of the liquor below 0·4 m./sec. the accelerating effect is drastically reduced. Both lofty and dense fabrics are amenable to these treatments. The nitrogenous impurities in cotton are not removed very effectively by the pressure method, but the products are suitable for dark dyeings. To reduce the nitrogen content a brief (15–20 min.) alkaline boil is suggested followed by hypochlorite bleaching. C. O. C.

Oxidation Potentials of Hypochlorite Solutions. B. P. Nikolsky and I. E. Flis. *J. Gen. Chem. U.S.S.R.*, **22**, 1298–1307 (Aug. 1952).

In hypochlorite solutions of pH $> 9{\cdot}3$, the observed oxidation potential at a Pt electrode is given (in volts) by the equation—

$$E = 1{\cdot}267 - 2{\cdot}3\frac{RT}{F}\left(\mathrm{pH} - 0{\cdot}5 \log \frac{a_{OCl^-}}{a_{Cl^-}}\right)$$

corresponding to the equilibrium—

$$OCl^- + 2H^+ + 2\ominus \rightleftharpoons Cl^- + H_2O$$

At pH < 3·5, the electrode behaves as a chlorine electrode, but in the intermediate pH range the potential, which is 55–60 mv. higher than that calculated by the above equation, is not in accord with any of the schemes that have been proposed; in particular, it is not affected by variation in $[Cl^-]$ and cannot be determined by the above equilibrium, the analogous equilibrium involving undissociated HOCl, or reactions involving the liberation of oxygen (the oxygen electrode potential is considerably lower than that observed). The addition of ClO_3^- ions has no effect on the potential over the pH range 5–10.

A. E. S.

Spectrophotometric Analysis of Acidified Sodium Chlorite Solutions. W. Buser and H. Hänisch. *Helv. Chim. Acta*, **35**, 2547–2556 (Dec. 1952).

Absorption spectra in the region 220–480 mμ. of ClO_2 and ClO_3^- in aq. soln. were determined. Alkali-metal chlorides and sulphates do not absorb at wavelengths > 200 mμ. nor ClO_3^- at > 240 mμ. The spectrum of $HClO_2$ could be derived theoretically from curves obtained in the presence of ClO_3^-, the absorption due to the latter being calc. with the help of the dissociation constant of the former. The stability of sodium chlorite in aq. soln. at different pH was examined by measurement of the intensity of absorption at 279 and 439 mμ., ClO_2 absorbing equally at both. The concn. of ClO_2 was calc. from the intensity at 439 mμ., where it alone absorbs. At 279 mμ. the absorption was due to ClO_2, $HClO_2$, and ClO_3^-. The part due to ClO_2 was known from the measurement at 439 mμ., and the ratio of the concn. of $HClO_2$ and ClO_3^- was calc. from the dissociation constant as above. The concn. of each species was thus derived. The rate of formation of ClO_2 in dil. chlorite soln. was no simple function of pH, but was very small at pH > 4. In the region pH 2–4, the ratio of ClO_2 formed to consumption of ClO_3^- and H^+ corresponded to the equation—

$$4\,ClO_3^- + 2\,H^+ \rightarrow 2\,ClO_2 + ClO_3^- + Cl^- + H_2O$$

At pH < 2 the ratio of ClO_2 formed to ClO_3^- consumed decreased. Graphs are given for the rate of formation of ClO_2 at pH 0–4.

H. E. N.

Buffering Bleach Solutions. I. E. Flis and E. Y. Perlshtein. *Bumazh. prom.*, **27**, (7), 16–19 (1952): *Chem. Abs.*, **46**, 11,678 (25 Nov. 1952).

The use of CaO–MgO^* to buffer hypochlorite soln. is discussed, and data are given from a number of bleach works; e.g. in a 4-hr. hypochlorite pulp-bleaching at 41°c. and pH 10·5, consumption of CaO was 18 kg./ton bleached pulp, the viscosities of the unbleached and bleached pulps were 1551 and 423 millipoises, and α-cellulose, ash, resins, breaking length, and brightness values for the bleached pulp were 87·6%, 0·26%, 0·65%, 6094 m., and 91 respectively.

C. O. C.

Equilibria in Bleach Liquor. I—Activity Constant of Hypochlorous Acid and its Variation with Ionic Strength. R. W. Asmussen and L. T. Muus. *Trans. Danish Acad. Tech. Sci.*, (1), 3–24 (1946): *Chem. Abs.*, **46**, 10,804 (25 Nov. 1952).

Study of the activity constant of HOCl by electrometric titration with a glass electrode gave $2{\cdot}8 \times 10^{-8}$ at 20°c. and $2{\cdot}2 \times 10^{-8}$ at 0·6°c. with a standard error of 0·3 in each. It was assumed that the activity coefficient of HOCl is unity, and that of OCl^- is given by the Debye–Hückel relation and is borne out by extrapolation of the acidity constant to zero value of $(1/f) - 1$. The ionic strength was varied by addition of Na_2SO_4 to a max. of 0·667 M.

C. O. C.

Influence of Bleaching on the Opacifying Properties of Pulp. H. W. Giertz. *Svensk Papperstidning*, **55**, 897–900 (15 Dec. 1952).

In bleaching, lignin and some of the hemicelluloses are dissolved, giving rise to light-scattering spaces in the fibre wall and leading to an increase in the light-scattering coefficient of the pulp. The improvement in brightness is, however, accompanied by a decrease in opacity, measured as printing opacity or contrast ratio. Oxidation of the hydroxyl groups of the cellulose or hemicellulose does not influence the light-scattering properties, but if the fibre structure is weakened during bleaching, the paper becomes denser and less opaque, particularly after beating and if high pressure is used during wet pressing. Consequently chlorine dioxide gives more opaque bleached pulps than hypochlorite.

S. V. S.

PATENTS

Continuous Bleaching of Cotton. Diamond Alkali Co. *BP* 683,493

The grey goods are treated with a hypochlorite soln. of available Cl content 0·3–4·5 g./litre kept at pH 7·0–9·5 and containing a wetting and penetrating agent. They are then treated with 0·5–3·0% aq. caustic alkali and again treated with hypochlorite as before. The goods are treated in each bath for ca. 1 min., and are stored, e.g. in a J-box, for not more than 1 hr. between each treatment. A full bleach is obtained in 3 hr. or less, and the results judged on whiteness and tensile strength are superior to those obtained by peroxide bleaching.

C. O. C.

Continuous Hypochlorite Bleaching of Cotton Cloth. Allied Chemical & Dye Corpn. *USP* 2,602,723

Highly bleached cloth can be continuously produced without much degradation by passage through a desizing bath, caustic treatment in direct contact with steam, and multistage bleaching with dil. aq. hypochlorite at low temperatures for short periods of time.

C. O. C.

Bleaching Chemical Pulp. Buffalo Electro-Chemical Co. *BP* 684,742

High-strength superbleached sulphate or sulphite pulps are obtained by impregnating the partly bleached pulp of brightness 65–85 (as measured in a standard brightness meter) and density > 30% with alkaline hydrogen peroxide and allowing to stand for 1–20 days at < 130°F. until maximum brightness is obtained, the density of the pulp being maintained at > 30% on a dry pulp basis.

C. O. C.

Mechanism of the Decomposition of Hydrogen Peroxide in Certain Solid Perhydrates. (III p. 88).

VIII—DYEING

Studies in Hydrogen-bond Formation. I—Detection of the Interaction of Phenols with Amido and Azo Groups, etc. by Dielectric-constant Measurement. C. H. Giles, T. J. Rose, and D. G. M. Vallance. *J.C.S.*, 3799–3805 (Oct. 1952).

Hydrogen bonding between organic compounds is readily detected by dielectric-constant measurement of binary solutions in anhydrous solvents, and it is found that phenolic hydroxy groups have a consistent tendency to bond with other similar groups or with nitrogen atoms in azo, amino, and secondary alkyl- and aryl-amido groups, but not with a keto group. Cross-linking of azo and alkylamido groups by dihydric phenols (resorcinol, quinol) has been detected. Two azo groups appear to be cross-linked by only one dihydric phenol molecule, whereas two alkylamido groups appear to be cross-linked by two dihydric phenol molecules in parallel. The amide group seems to react in the enol form and is bifunctional.

H. H. H.

Direct Dyeing of Cellulose. I—Construction of a Photoelectric Colorimeter and Direct Dyeing. K. Nishida. *J. Soc. Textile Cellulose Ind.* (Japan), **8**, 347–350 (1952): *Chem. Abs.*, **46**, 9851 (25 Oct. 1952).

The isotherms for the absorption of Diamine Blue 2 B (0·01–0·05 g./litre) by Cellophane and oxycellulose were determined in a specially made photoelectric colorimeter having a gas-filled selenium cell. With > 5 g. NaCl per litre of dye liquor the curves were typically parabolic or hyperbolic, but became S-shaped with larger amounts of NaCl. The initial slow absorption was explained by assuming that the cellulose carboxylic acid (oxidised from Cellophane) dissociated between the micelles into cellulose-COO^- and H^+ (replaced in practice by a metal ion, e.g. Na^+) and was stopped from approaching the direct dye ($R^- + Na^+$) by their mutual electrical repulsion. As more NaCl is added, absorption increases, as the less dissociated cellulose-COOH can approach the dye. This is supported by the fact that the S-shape became more pronounced when the Cellophane was first oxidised by $KMnO_4$, and still more pronounced when no NaCl was present.

C. O. C.

Relation between Tinctorial Affinity and Degree of Swelling of Acetylated Cellulose. C. Mayrargue and R. A. Pâris. *C. R. Acad. Sci. Paris*, **234**, 2452–2454 (1952): *Chem. Abs.*, **46**, 10,612 (10 Nov. 1952).

The affinity of acetylated cellulose (acetyl value 0–52·5%) for direct dyes passes through a maximum corresponding to that of the water adsorption of the sample. Undried viscose rayon, acetylated with increasing amounts of acetic anhydride at room temp. with H_2SO_4 as catalyst, was rinsed with cold ethyl alcohol, stabilised by boiling for 4 hr. in aq. ethyl alcohol, and dried. The affinity of the acetylated fibre for Direct Sky Blue Green Shade, Chlorantine Brilliant Orange, and Benzopurpurin was determined colorimetrically, and their water absorption by the Champetier method (*Chem. Abs.*, **27**, 5183 (1933)). Fixation of direct dyes by sufficiently swollen fibre is proportional to the degree of swelling. C. O. C.

Metachromatic Effects of Anionic Polysaccharides and Detergents. A. Levine and M. Schubert. *J. Amer. Chem. Soc.*, **74**, 5702–5706 (20 Nov. 1952).

Quantitative comparison of the chromotropic action of six anionic polysaccharides and two anionic detergents on two cationic metachromatic dyes of different structures showed that all of them produced effects showing similar trends on the α-, β-, and μ-bands of both dyes, there being quantitative differences between the effects on each dye. It seems that methylene blue and crystal violet, which are metachromatic, are bound by polysaccharides, whereas methylene green, which is hardly metachromatic, is bound to a much smaller extent, if at all. Presence of salt, which destroys metachromasy, also destroys the binding of the dyes by polysaccharides as measured by equilibrium dialysis, whereas high chromotrope concentration destroys metachromasy but not dye binding. C. O. C.

Light-fast Dyes from Tannin-extraction Residues. B. Ya. Rozen. *Tekstil. prom.*, **12**, 32–33 (Sept. 1952).

It is suggested that greater use could be made of the residues remaining after extracting tannin from vegetable materials. The residue from tamarisk is extracted with water or aq. alkali, and the dye soln. obtained is applied to wool and silk by one-bath mordant-dyeing processes. The dull colours obtained by the use of chrome, alum, and lead mordants are stated to be fast to light, washing, and perspiration. A. E. S.

Exact Pattern of a Concentration-dependent Diffusion in a Semi-infinite Medium.— I. H. Fujita. *Text. Research J.*, **22**, 757–760 (Nov. 1952).

A formal exact solution of a unidimensional diffusion in a semi-infinite medium in which the diffusion coefficient $D(c)$ varies with concentration c in the following manner—

$$D(c) = \frac{D(0)}{1 - \lambda c}$$

(λ = a parameter). A. B.

Rôle of Sodium Chloride and Sodium Sulphate in the Acid Dyeing of Wool. J. D. Justice and W. W. Ewing. *Amer. Dyestuff Rep.*, **41**, P 668–P 677 (13 Oct. 1952).

The dyeing properties of selected acid dyes, with different levelling powers, are studied using NaCl, with HCl or H_2SO_4, in place of the normal Na_2SO_4, at various pH values. A modified Gilbert–Rideal equation is presented, and good agreement is obtained between theoretical and practical results. Up to four times as much NaCl as Na_2SO_4 is necessary; the requisite quantity increases with decreasing initial pH, and is smaller at higher liquor ratios and also at lower initial dye concentrations. In general, the lower the affinity of the dye the smaller the amount of NaCl required for direct replacement of Na_2SO_4; with high-affinity dyes it is no more effective than Na_2SO_4, and, as usual, higher pH values are necessary to obtain level dyeing. J. W. B.

Interchangeability of Sodium Sulphate and Sodium Chloride in the Acid Dyeing of Wool. H. W. Leitch. *Amer. Dyestuff Rep.*, **41**, P 678–P 681 (13 Oct. 1952).

Laboratory and semi-bulk trials on the use of NaCl instead of Na_2SO_4 in wool dyeing are followed by a three-month full-scale works production trial. In general, 40% of NaCl on the wt. of fabric produces satisfactory results; in particular, one normally difficult recipe gave no trouble when NaCl was used. No corrosion was encountered with stainless steel equipment. J. W. B.

Theory and Practice of Wool Dyeing with Metal-(Chromium)-complex Acid Dyes. R. Casty. *Melliand Textilber.*, **33**, 950–954 (Oct. 1952).

The conditions to be observed when dyeing wool with commercial "1 : 1" chrome-dye complexes (e.g. Neolan dyes) are discussed. Increase in the concn. of sulphuric acid in the dyebath results in two mutually opposing tendencies— increase in dyeing rate associated with the presence in the dye molecule of sulpho groups, and decrease in dyeing rate due to salt formation with the amino groups of the wool, which then have a lower tendency to enter the chrome complex of the dye. Max. control of dyeing is reached at pH 2, when the wool has practically attained its max. uptake of H_2SO_4 (4 g. per 100 g. of wool), and it is recommended that this amount of acid together with the further amount required to establish the pH (0·3–1 g./litre) should be used in practical dyeing. Control can be effected in presence of a lower concn. of acid by adding non-ionic auxiliaries (e.g. Palatine Fast Salt O) that associate with the dye and reduce the concn. of available dye ions. Sodium sulphate is a useful restrainer for dyes containing two sulpho groups. Further practical information is given. A. E. S.

Cibalan Dyes on Wool. J. G. Grundy. *Dyer*, **108**, 685–692 (14 Nov. 1952).

This new range of premetallised dyes, comprising twelve members, offers advantages over existing combined metal types for wool insofar as they are applied from a neutral or weakly acid bath. Dyeing is best carried out at pH 6–7, and consequently alkaline water or alkaline wool requires the use of a buffer salt to maintain the correct pH; Glauber's salt is of no assistance and may cause dye agglomeration. Experimental evidence is given which shows that, of the three salts used — ammonium acetate, ammonium sulphate, and mono-ammonium phosphate— the acetate is the most suitable. Data in the form of graphs show temperature range and rate-of-dyeing experiments, and a table gives detailed fastness properties; light fastness is 6–7, and other fastness figures are also very good. Silk, nylon, and union dyeing are briefly discussed. A. H.

Hosiery Dyeing and Finishing. H. L. DeTurck. *Amer. Dyestuff Rep.*, **41**, P 739–P 743 (10 Nov. 1952).

A brief general survey of hosiery dyeing and finishing is followed by an account of procedures for nylon, Terylene, Perlon, and nylon–silk mixtures. Conventional and alcoholic stripping are discussed, together with some finishing techniques, particularly snag-resistant resin finishes. J. W. B.

Intensifying Aniline Black Dyeings. P. V. Gorshkov. *Ivanov. Nauch.-issledovatel. Inst. Khlopchatobumazh. prom., Nauch.-issledovatel. Trudy*, **18**, 100–108 (1951): *Chem. Abs.*, **46**, 9850 (25 Oct. 1952).

A study of the drying process. When drying with air at 50–100°c. using the usual soln. of 55 g. aniline salt, 25 g. $KClO_3$, 12 g. $CuSO_4$, and 10 g. NH_4Cl per litre of water, the cloth can be dried to 10% residual moisture without damage to the fibre. In cylinder drying the high temperature (150°c.) of the cylinder means that a higher moisture content (50%) must be left, and drying completed with hot air. The dyed cloth was air-dried and then treated for 7–20 min. to 0·3–1·5 min. at 50–100°c., fibre damage and general condition being noted. Best results were obtained in 10–12 min. at 50°c., 5–7 min. at 60°c., 1·5–2·0 min. at 80°c., and 0·3–0·35 min. at 100°c.; as expected, ageing of the dye is accelerated by increase in temperature, but fibre damage increases rapidly with increase in temperature. Fibre damage increases on heating in presence of high moisture content, as is shown by increase of the ferricyanide No.; pH tests show that this is caused by more HCl being present in the wetter cloths. Thus, a sample kept at 60°c. and 10–15% R.H. gave an extract of pH 2·6, while one in 70% R.H. gave an extract of pH 1·3. An experimental apparatus is described having two sets of heated cylinders, the first set drying to 50% moisture content and the second to 20%, followed by an air-drying chamber maintained at

80–85°c., after which there is another chamber in which the cloth is heated for 1·5–2·5 min. at 30% R.H. The cooled cloth is chromed to complete the dyeing. C. O. C.

Dyeing of Sulphur Dyes with Steaming. S. A. Plaksin and P. G. Vykhlyantsev. *Ivanov. Nauch.-issledovatel. Inst. Khlopchatobumazh. prom., Nauch.-issledovatel. Trudy*, **18**, 90–99 (1951): *Chem. Abs.*, **46**, 9850 (25 Oct. 1952).

Reduction by 33% of the Na_2S normally used and 10–20% better tinctorial value are obtained by passing the cloth for 30 sec. through the dyebath and then for 42 sec. through a specially designed rectangular chamber filled with steam at 98–100°c., from which air is excluded. Small amounts of Na_2S occur in the rinsing baths. C. O. C.

Dyeing Modern Synthetic Fibres. B. Kramrisch. *Dyer*, **108**, 709–713 (14 Nov. 1952).

Report of a lecture describing in a general way the dyeing procedures for Ardil, dynel, Terylene, Thermovyl, Rhovyl, Perlon L, Orlon, etc., A. H.

PATENTS

Increasing the Wet Fastness of Dyeings on Cellulosic Fibres. CFM. *BP* 683,251

Dyeings in which a water-soluble dye is applied to the fibre but is rendered less soluble by treatment on the fibre, e.g. sulphur dyes or diazotised and developed direct dyes, are given improved wet fastness by treatment with a peralkylation product of a polymerised alkyleneimine, e.g. the product obtained by the exhaustive methylation of an ethyleneimine polymerisate with dimethyl sulphate. C. O. C.

Metachrome Dyeing. American Cyanamid Co. *BP* 683,761

If a cationic surface-active agent is being used as an assistant in metachrome dyebaths, improved results are obtained if the dyebath is set at a pH high enough to prevent or retard rapid chromation. This pH varies with different dyes. With many dyes deeper dyeings can be obtained, while others inapplicable by normal metachrome methods can be satisfactorily dyed. There is also a considerable saving in chromium salt, in some cases as much as 50%. C. O. C.

Dyeing Cellulose Acetate. Celanese Corpn. of America. *USP* 2,601,460

Moulded articles having a basis of cellulose acetate or other organic derivative of cellulose are very rapidly dyed deep level colours which are fast to rubbing by using a bath containing an aqueous soap dispersion of a dye for the cellulose acetate, and a water-soluble organic solvent for the dye, the solvent being preferably one of formula $R{\cdot}O{\cdot}[CH_2{\cdot}CH_2{\cdot}O]_n{\cdot}CH_2{\cdot}CH_2OH$ (R = lower alkyl; n = 0 or 1), e.g. β-butoxyethyl alcohol. C. O. C.

Increasing the Dyeing Affinity of Terylene. ICI. *BP* 684,046

Heating at 230–255°c. before dyeing markedly increases the affinity of Terylene for dyes. Preferably heating is effected by contact with a hot metal surface. Yarns which have been highly oriented by drawing before heating are given good textile properties if they are allowed to relax during heating. C. O. C.

Improving the Dyeing Properties of and Finishing Terylene. Calico Printers' Assocn. *BP* 683,218

Treatment with 70–90% aq. H_2SO_4 or chlorosulphonic acid at − 10 to 110°c. confers greatly improved affinity for many dyes. The treated material also dyes and prints more evenly and much more rapidly than the untreated material. In addition to improving the dyeing properties, acid of strength 85% or more imparts stiffening or parchmentises the material, whereas at strengths of 77% or less delustring occurs.

BP 683,634

Treatment with 30–78% aq. HNO_3 at −10 to 110°c. similarly affects the affinity for dyes. Concentration of acid, temperature, and duration of treatment are interdependent in securing the same alteration of dyeing properties. The material is also shrunk, a shrinkage of up to 45%, accompanied by extremely high dye affinity, being obtained with 76% acid at − 10 to + 10°c. C. O. C.

Sonic Vibration as an Aid in Dyeing. British Thomson-Houston Co. *BP* 683,105

If the material is vibrated at 25–1000 cycles/sec. with a peak-to-peak vibrating displacement of the material of > 0·05 in., considerably better penetration is obtained even though the vibration is produced by relatively low-power mechanism. C. O. C.

Emulsions for Colouring Fabrics. American Cyanamid Co. *USP* 2,601,661

Oil-in-water emulsions, in which the dispersed oil is volatile at the setting or curing temperature used for the film-forming substance present, in which the oil makes up 4·5–70% of the emulsion, and in which the film-forming constituent and colouring matters are dissolved or dispersed in the aqueous phase, when applied to fabric yield colorations without a stiff handle, the oil preventing formation of a continuous film which might bridge the interstices between the fibres. C. O. C.

New Problems Encountered by the Garment Dyer and Cleaner (VII p. 100).
Alaska Sealskins — Fur Seal into Fur (XII p. 106).

IX— PRINTING

Problems in Screen Printing with Vat Dyes. II— Steaming. G. Bernardy. *Melliand Textilber.*, **33**, 865–867 (Sept.), 954–957 (Oct. 1952).

Continuing the discussion of the screen printing of vat dyes by the formaldehyde-sulphoxylate method (see J.S.D.C., **68**, 416 (Oct. 1952)), various types of ager are described, and the general precautions that must be taken with respect to the condition and method of application of the steam, particularly in star agers, are detailed. In continuous steaming, marking-off is largely avoided by the use of the "tower" ager, in which the face of the fabric comes into contact only with the entry roller. The advantages of the Colloresin method for direct printing are again emphasised. A. E. S.

PATENTS

Water-in-lacquer Emulsions for Application to Textiles. ICI. *USP* 2,600,890

Very stable water-in-lacquer emulsions are produced by use of alkyd resins obtained by reaction of an adduct of an α-unsat. acid and a terpene with a partial ester of pentaerythritol with the copolymer of styrene and dehydrated castor oil. Prints made with such an emulsion have improved fastness to washing and excellent resistance to the action of alkali. C. O. C.

Printed Relief Effects. A. I. A. Versteeg. *BP* 684,696

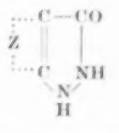
The patterns or at least part of them are built up of dots and/or short lines or blocks in the manner of a print obtained by screen printing with a screen whose mesh size is at least 1 mm. This gives a relief effect when the patterns are viewed from a distance. C. O. C.

Fixing Mechanically Produced Effects. Calico Printers' Assocn. *BP* 684,849

Those parts of the fabric which are to be left plain are treated with an aqueous solution of a salt which readily becomes insoluble when heated but remains capable of neutralising an alkaline catalyst. The fabric is then thoroughly heat-dried, and impregnated with an aqueous solution of a precondensate of an aldehyde with another aldehyde, ketone, aldehyde-alcohol, or ketone-alcohol, and an alkaline catalyst. It is then dried at moderate temperature, conditioned if necessary, and given the desired mechanical finish, baked to cure the resin, and washed. C. O. C.

Multicolour Photographs. Gevaert. *BP* 685,061

A complementary image is formed by use of a colour coupler whose residue after colour development is oxidised to a coloured substance, e.g. compounds of formula—



(Z = atoms to complete an isocyclic or heterocyclic ring). C. O. C.

Colour Photography. ICI. *BP* 685,607

Use in the original photographic record of an unnatural-order combination together with a black and white mask in at least one of the cyan and magenta layers to increase the saturation of the colours results in high colour fidelity in the positive.

BP 685,608

With dyes of unequal undesired absorptions the duplicate record which can be obtained from an unnatural-order original photographic record by a reversal process gives positive prints much superior in colour fidelity to those from a duplicate negative prepared by a reversal process from a simple natural-order original negative.

BP 685,609

Combination of the two above processes gives positives of higher colour saturation as well as high colour fidelity.

BP 685,610–1

The black and white mask is replaced by an azo masking image in the cyan and/or magenta layers.

BP 685,612

The black and white mask of *BP* 685,607 (above) is replaced by a yellow styryl dye masking image on the magenta layer.

BP 685,613

Still better colour fidelity is obtained by the process of *BP* 685,608 (above) if the duplicate record is treated so as to form a yellow styryl dye mask on the magenta layer.

C. O. C.

X—SIZING AND FINISHING

Stripping of Synthetic Condensation Resins. E. Schönpflug and H. Anschütz. *Melliand Textilber.*, **33**, 959–960 (Oct.), 1036–1038 (Nov. 1952).

Staple rayon fabrics finished with synthetic resins formed by condensation within the fibre, viz. Kaurit KF (urea–formaldehyde) and Cassurit MKF (melamine–formaldehyde), are treated in various alkaline and acid baths, and the degree of resin removal is determined by direct weighing and by testing for residual formaldehyde. Tensile properties of the fabric are determined before and after stripping. For the urea resin, the best stripping treatment is 20 min. at 70°c. at a liquor ratio of 30 : 1 in a soln. containing, per litre, 2 ml. of 85% formic acid and 2 g. of Levapon TH; for the melamine resin the best stripping liquor contains, per litre, 4 ml. of conc. HCl and 2 g. of Lavagent NA, the conditions being otherwise the same. The change in fibre strength during stripping is negligible. A. E. S.

Improved Comfort by Reducing the Coefficient of Friction of Wet Vegetable Fibres. C. Normand. *Industrie textile*, **69**, 271–273 (1952): *Chem. Abs.*, **46**, 11,691 (25 Nov. 1952).

The large friction of wet cotton, linen, and rayon affects the comfort of the wearers of such materials. This disadvantage is eliminated by urea–HCHO finishes containing sulphates, chlorides, nitrates, or acetates of Zn, Cd, Mn, or Cu to buffer the system at pH 6·0–6·5. C. O. C.

Permanent Set of Keratin Fibres. I—Comparison of Setting Properties of Various Keratin Fibres. S. Okajima. *J. Soc. Textile Cellulose Ind.* (Japan), **6**, 330–334 (1950); **II—Some Problems of the Water required for the Permanent Set.** *Ibid.*, 460–464; **III—Effect of Permanent Set on the Mechanical Properties of Keratin Fibres.** *Ibid.*, **7**, 381–384 (1951): *Chem. Abs.*, **46**, 10,627 (10 Nov. 1952).

I—The permanent set p, calculated from $p = (L/L_0) - 1$ (L_0 = initial straightened length; L = length after stretching a known amount with a brass rack in a buffer (pH 7·9–10·1), boiling for 30 min., and then releasing and boiling again for 30 min.) differed hardly, if at all, between merino wool, mohair, and cashmere; it increased with stretching. Camel's hair gave a poor set.

II—Natural water in large amount, especially when boiled, could be used as the setting medium, as the $CaCO_3$ and $MgCO_3$ contents kept the pH within the optimum range. When the fibres were pretreated with NaOH, the volume of water required was kept to a minimum by steam setting.

III—Steaming for 30 min. did not affect unstretched wool but lowered the tensile strength of stretched fibres. Before setting the elongation increased with stretching in steaming. A serge sample was slightly damaged by steaming owing to stretching of the fibres during spinning and wearing. C. O. C.

Polyvinyl Acetate and its Derivatives as a Basis for Textile Auxiliaries. W. Starck. *Melliand Textilber.*, **33**, 944–946 (Oct. 1952).

A description is given of the preparation of vinyl acetate polymers and copolymers in mass, in soln., and as latices and of their further partial or complete conversion to a vinyl alcohol (co)polymer, which may be treated with aldehydes to give further products. The application of all these materials in textile and other fields is discussed. A. E. S.

Theory of Water-resistant Fabrics. G. H. Segall. *Text. Research J.*, **22**, 736–741 (Nov. 1952).

It is shown that the water-resistance of a fabric depends on the hydrostatic pressure resistance of the pores of the fabric and the interfibre penetration pressure of the fabric pore walls. When these correspond at a small pore size a high degree of water resistance will be obtained. A. B.

Water-resistant Textiles. S. J. Kennedy. *Quarter Master Research and Development Laboratories* (*U.S.*), *Textile Report Series* No. 37* (PB 105,661). Pp. 196. Price, Microfilm 49s. 0d.

A summary of fundamental and applied research carried out between 1940 and 1947 on the water-resistance of textiles. Some of the many aspects dealt with include the physics of wetting, the effect of fabric structure, and water-repellent finishes and their durability. Test procedures are described. C. O. C.

*Photocopies available from T.I.D.U., D.S.I.R., Cunard Building, 15 Regent Street, London S.W.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see J.S.D.C., **66**, 53 (Jan. 1950).

Degradation from Weathering of Tent Cloths. W. E. C. Yelland. *Quarter Master Research and Development Laboratories* (*U.S.*), *Textile Report Series* No. 40* (PB 105,767). Pp. 156. Price, Microfilm 39s. 0d.

Details of an investigation of the weathering resistance of a 12·29 oz. duck exposed in temperate and semi-tropical climates. Mineral-dyed grey and fire-, water-, weather-, and mildew-resistant finishes were tested. Degradation was due to hydrolysis and oxidation of cellulose. Short-wave ultraviolet radiation was most effective in initiating degradation. C. O. C.

*Photocopies available from T.I.D.U., D.S.I.R., Cunard Building, 15 Regent Street, London S.W.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see J.S.D.C., **66**, 53 (Jan. 1950).

Behaviour of Nylon Oxford Cloth "41" on Pyrolysis. T. Hasselstrom, H. W. Coles, C. E. Balmer, M. Hannigan, M. M. Keeler, and R. J. Brown. *Text. Research J.*, **22**, 742–748 (Nov. 1952).

It is shown that pyrolysis of nylon fabric at temp. of 280–400°c. causes depolymerisation of the polymer, and not cross-linking of the aliphatic polymer chains. Considerable amounts of ammonia are formed during pyrolysis. Most conventional flameproofing chemicals tend to increase the flammability of nylon, with the exception of certain sulphur- or chlorine-containing compounds. A. B.

Flameproofing of Fabrics—A Literature and Patent Survey. F. D. Horigan and C. R. Sage. *Quarter Master Research and Development Laboratories* (*U.S.*), *Bibliographic Series* No. 18* (PB 105,479). 50 pp. Price, Microfilm 12s. 6d.; Enlarged Prints 50s. 0d.

211 brief abstracts. Many of the treatments dealt with are fungicidal and water-repellent. C. O. C.

*Photocopies available from T.I.D.U., D.S.I.R., Cunard Building, 15 Regent Street, London S.W.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see J.S.D.C., **66**, 53 (Jan. 1950).

PATENTS

Hydroxymethyl-dimethylhydantoin for rendering Cellulose Fabrics dimensionally Stable and Crease-resisting. L. Beer. *USP* 2,602,017–8

Dimensional stability and crease resistance are obtained, without loss of tensile strength and without imparting a tendency toward chlorine retention, by impregnation with an aqueous solution of hydroxymethyl-dimethylhydantoin in presence of a catalyst which gives the solution pH 2·0–2·5, followed by drying at 80–100°c. and baking at 120–150°c. C. O. C.

Chlorinating Wool. Scholler Brothers. *BP* 683,762
Accurate control of chlorination, uniform treatment, and reduction in yellowing, harshening of handle, and alkali solubility are obtained by treating the wool so that it retains 0·49–0·55 m-equiv. of acid per gram of air-dried wool and then treating for $\leq$ 5 min. in a chlorinating bath at pH 7·2–9·0 in presence of an alkaline borate buffer.
C. O. C.

Setting Twist or Curl in Keratinous Fibres. Monsanto. *BP* 685,201
The material is treated with a liquid containing an amino–aldehyde thermosetting condensate and a mercapto-alcohol, mercapto-polyalcohol, or aliphatic mercapto-carboxylic acid or salt thereof, and is then heat-cured.
C. O. C.

Stiff and Resilient Felts of Good Wet and Dry Strength. Monsanto. *BP* 685,202
Felt containing keratinous fibres is given improved wet and dry strength, resistance to shrinkage, water absorption, and natural resilience and stiffness by treating it with a liquid containing a condensation product and a mono- or poly-hydric aliphatic mercapto-alcohol and then heat-curing.
C. O. C.

Stabilising and Setting Tubular-knitted Nylon Fabrics. Adshead & Geeson. *BP* 683,075
The fabric is passed through steam to obtain extensibility, then through hot dry air, and finally over and in contact with a surface at $\leq$ 300°F.
C. O. C.

Nylon Setting. British Nylon Spinners. *BP* 684,837
The fabric with its dimensions under mechanical control is immersed in mercury at a temperature between 160°C. and that 25°C. below the m.p. of the nylon. Other conditions being equivalent, this gives a better set than that obtained by gaseous setting.
C. O. C.

Eliminating Knot Slippage in Fishing Nets. British Nylon Spinners. *BP* 685,623
Nets are made from a synthetic linear polyamide containing CO·NH groups, and the knots are secured by applying to the yarn, before or during knotting, an adhesive comprising a soln. made by dissolving, with heat if necessary, a synthetic linear polyamide containing CO·NH groups in a mixture comprising an aliphatic alcohol boiling at $<$ 200°C., formaldehyde, and an unsubstituted liquid fatty acid and/or a mixture of an aliphatic hydroxy acid and formaldehyde, with or without water, and removing solvent by evaporation so that the adhesive hardens.
J. W. B.

Water-repellent Finish. National Lead Co. *BP* 684,686
The material is impregnated with a zirconium compound containing Zr in a complex carbonato anion, a fatty acid compound, and, if desired, an emulsion wax, and then, before drying, is treated in an aqueous solution of a salt of a metal of Group III, IV, or VIII, e.g. a soluble Zr, Al, or Sn salt in which the metal is present as cation. This renders the finish more resistant to washing and dry cleaning.
C. O. C.

Suedette Material. Dunlop Rubber Co. *BP* 684,708
Backing material is coated with a plasticised vinyl chloride polymer or copolymer which may be converted by heat into a porous gel, textile fibres are distributed over this coating, and the whole is heated so as to gel the polymer and anchor the fibres in it.
C. O. C.

Hosiery Dyeing and Finishing (VIII p. 102).
Increasing the Dyeing Affinity of Terylene (VIII p. 103).
Improving the Dyeing Properties of and Finishing Terylene (VIII p. 103).
Emulsions for Colouring Fabrics (VIII p. 103).
Fixing Mechanically Produced Effects (IX p. 103).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Degradation of Cotton and Wood Pulp with Lignin-sulphonic Acid and Sulphite Waste Liquor. N. Hartler and O. Samuelson. *Svensk Papperstidning*, **55**, 851–862 (30 Nov. 1952).
A study of the hydrolysis of cellulose fibres with sulphuric acid, high-molecular ligninsulphonic acid, and sulphite waste liquor showed that, at acid concentrations giving the same inversion velocity of cane sugar, an appreciably slower degradation of the cellulose fibres was obtained with ligninsulphonic acid than with sulphuric acid. The fractions of the cellulose fibres that are accessible to the low-molecular acid are not penetrated by the high-molecular. Although sulphite cooking takes place in a mixture of high- and low-molecular acid, the course of degradation is that due to a low-molecular acid, which can be explained by the Donnan theory. Cotton and wood pulp show differences, e.g. in the viscosity of the cellulose as a function of time of hydrolysis, the curves crossing one another twice.
S. V. S.

Infrared Spectra of Bacterial Cellulose. H. G. Shirk and G. A. Greathouse. *Anal. Chem.*, **24**, 1774–1775 (Nov. 1952).
Close similarity between the infrared spectra of pellicle membranes produced by *Acetobacter xylinum* from various compounds such as cellobiose, mannitol, glycerol, dextrose, and lævulose indicates that they are all essentially the same in structure as higher-plant cellulose. Small variations in absorption intensity and definition, particularly in the 7·0–8·5 μ. region, are attributed to differences in film thickness. The synthesis is unaffected by the presence of radioactive carbon atoms in the substrate used.
J. W. D.

Electron Microscopy of Degraded Cellulose Fibres. Criticism by H. W. Emerton and P. E. Wrist; Reply by J. Sikorski and H. J. Woods. *J. Textile Inst.*, **43**, T 563–T 564 (Nov. 1952).
It is suggested that the inference by Mukherjee, Sikorski, and Woods (*ibid.*, **42**, T 196 (April 1952)) that the oriented structure of lightly beaten esparto grass changes on heavy beacing to a highly oriented reticulate structure is untenable, and that the correct interpretation is that one photograph represents a secondary, and the other a primary, wall fragment.
In reply, it is emphasised that this aspect is of minor importance and that it was the intention to contrast the fibrillar type of breakdown caused by mechanical action with the particulate type found in acid-degraded fibres. Even so, it is considered that mechanical action can, in fact, produce pseudo-reticulates from normal fibrous structures.
J. W. B.

PATENTS

Coating Cellulosic Fibres with Dispersions. American Cyanamid Co. *USP* 2,601,597–8
Addition of a cationic alkylenepolyamine–halogenohydrin or urea–formaldehyde resin to a suspension of paper pulp and water-insoluble coating or impregnating agents causes a controlled flocculation, so that the agent is uniformly coated on or impregnated in the cellulose fibres. The fibres retain their felting property despite the presence of even more than their own weight of coating or impregnating agent.
C. O. C.

Thermosetting Resins for Producing Paper of High Wet Strength. Monsanto. *USP* 2,601,665
The product obtained by interaction of an amino compound of 1–6 C containing the grouping $NH_2{\cdot}CZ{\cdot}N<$ (Z = O, S, or N–), an aldehyde, and a water-soluble salt of a nuclear-substituted aromatic sulphonic acid of 6–10 C is added to the pulp in the beater.
USP 2,601,666
The resin is made by interaction in an alkaline aqueous solution of urea, formaldehyde, and an amino aliphatic mono- or di-carboxylic acid of 1–6 C or a salt or addition compound of such acid. The resulting solution is concentrated to 55–65% by wt. of resin solids, and is then made acid, preferably with a mineral acid. It is then allowed to condense and polymerise till a 60% by wt. soln. of the resin has a viscosity of 5–10 poises as measured by the Gardner–Holt bubble viscometer. The soln. is finally made alkaline to prevent further condensation.
C. O. C.

Drying Loosely Matted or Felted Web. H. Haas. *BP* 684,738
Webs containing agglomerations or "clots" of fibres can be satisfactorily dried without damage by first passing through successive zones alternately of suction and pressure to dry the normal part of the webs and then through a high-frequency electric field to dry and loosen the fibres in the clots.
C. O. C.

Pigment-coated Papers. Dow Chemical Co. *BP* 684,995

A paper-coating composition consists of a mineral pigment (100 parts by weight), e.g. clay, and an adhesive (8–30). The adhesive consists of an aqueous dispersion of casein, starch or soya protein (1), and a butadiene–styrene latex (0·333–3·0). A coated printing paper is made by applying a layer of the coating mix described and subsequently drying it. S. V. S.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Combination of Cation-active Compounds with Collagen and Chrome-tanned Collagen. D. A. Plant. *J. Soc. Leather Trades Chem.*, **36**, 351–357 (Nov. 1952).

The combination of cetyltrimethylammonium bromide with collagen and chrome-tanned collagen is studied at varying pH values. The curves obtained are very different, and indicate that free or non-zwitterion carboxyl groups are blocked by chrome tannage; it is not possible to determine to what extent zwitterion carboxyl groups are involved, as there is lack of stoichiometry in the combination above pH 9, where back titration of the zwitterions commences. J. W. B.

Alaska Sealskins—Fur Seal into Fur. W. Born. *Ciba Rev.*, **8**, (94), 3372–3394 (Oct. 1952).

Accounts of the Pribilof Islands, the home of the Alaska fur seals, and sealing on the Pribilofs are followed by a brief history of the sealskin-dyeing industry and an illustrated account of the dressing and dyeing processes now used in the U.S.A. C. O. C.

Infrared Dichroism and Protein Structure. R. D. B. Fraser and W. C. Price. *Nature*, **170**, 490–491 (20 Sept. 1952).

Assumptions that the principal absorption bands observed in the infrared spectra of proteins arise from highly localised bond-stretching or bond-deformation vibrations, so that the associated transition moment directions are along or perpendicular to the bonds according to the type of vibration involved, are considered inadequate. An approximate mathematical treatment of the normal vibrational modes of the peptide group is presented, and the dichroism to be expected from an oriented polypeptide in which the chains have a 3·7-residue helix configuration is calculated. Results are close to the experimentally observed values for poly-γ-benzyl-L-glutamate in the folded configuration, and it is considered, therefore, that the observations are in good agreement with the existence of a Pauling–Corey 3·7-residue helix. J. W. B.

New Method for Acetylating Proteins. J. Baddiley, R. A. Kekwick, and E. M. Thain. *Nature*, **170**, 968–970 (6 Dec. 1952).

Acetylthiols are capable of acetylating amino compounds, and 2-acetylthioethylacetamide—

$$CH_3{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}CH_2{\cdot}S{\cdot}CO{\cdot}CH_3$$

is used for preparing partly acetylated proteins. It is soluble in water, stable towards dilute acids and alkalis, and readily acetylates amines. In one example an almost quantitative yield of acetylglycine is obtained when the reagent (2 mol.) is added to a 10% soln. of Na glycine (1 mol.) and the soln. kept at 37°C. for 12 hr. With alanine, 3–4 days are required. J. W. B.

Spectrophotometry of Biuret Complexes as a Method of Investigating Proteins and Peptides. XIV—Effect of the Amino Acid Composition of a Polypeptide on the Absorption Spectrum of its Biuret Complex. M. I. Plekhan. *J. Gen. Chem. U.S.S.R.*, **22**, 1633–1644 (Sept. 1952).

With the view of relating the absorption spectrum of the cpd. formed by adding a Cu salt to an alkaline soln. of a protein (the "biuret complex") to the amino acid composition of the protein, various di- and tri-peptides are synthesised and their complexes examined spectrophotometrically. It is considered that in the complex the Cu is covalently linked to six atoms, of which 2, 3, or 4 may be N atoms, the remainder being O atoms. The 2N-complexes (blue) have absorption max. at 615–670 mμ. (e.g. glycylglycine), the 3N-complexes (violet) have absorption max. at 540–580 mμ. (e.g. glycylasparagine, diglycylglycine), and the 4N-complexes (red) have absorption max. at 515–535 mμ. (e.g. leucylglycylasparagine). A. E. S.

Behaviour of a Synthetic Polypeptide analogous to Protein Denaturation. A. Elliott. *Nature*, **170**, 1066–1067 (20 Dec. 1952).

It is suggested that, in certain respects, there is a considerable resemblance between poly-DL-alanine and the soluble form of silk obtained from solution in cupriethylenediamine. Poly-DL-alanine, of degree of polymerisation about 500, is readily soluble in cold water, giving a mobile solution from which coherent films may be cast; the infrared spectrum indicates a folded polypeptide chain. Heating leads to separation of a gel, which is not redissolved on cooling; and on keeping the solution at room temp. for several weeks a gel is formed, the spectrum of which shows signs of an extended (β) polypeptide chain. This gel dissolves in formic acid, and a film cast from this solution is water-soluble and shows only the folded polypeptide spectrum, indicating that the polypeptide has been taken through a cycle back to its original condition. Such a readily induced $\alpha \rightarrow \beta$ change, reminiscent of denaturation and renaturation of proteins, indicates a very nice balance between the α and β forms. It is also found that the polymer dissolves less readily in D_2O than in H_2O; no explanation of this is suggested. J. W. B.

Configuration of the Peptide Link and of Asparagine in Glycyl-L-Asparagine. L. Katz, R. A. Pasternak, and R. B. Corey. *Nature*, **170**, 1066 (20 Dec. 1952).

A three-dimensional X-ray analysis of the structure of glycyl-L-asparagine is employed to derive precise dimensions of the peptide link. A diagram is presented in which the dimensions agree with those postulated by Pauling and Corey, particularly with regard to their assumption of planarity for the peptide group. J. W. B.

Synthesis of Peptides. B. L. Shapiro. *Chem. and Ind.*, 1119–1125 (15 Nov. 1952).

A review article describing and discussing (*a*) syntheses which, following the classical work of Fischer, proceed through linear intermediates by condensation reactions and (*b*) syntheses involving cyclic intermediates by way of addition reactions. Polypeptides of high molecular weight are considered only briefly. 81 references. W. R. M.

PATENT

Resin-impregnated Leather Board. American Cyanamid Co. *USP* 2,601,671

Addition of a cationic melamine–aldehyde resin to an aqueous dispersion of leather fibres and water-insoluble coating or impregnating agents causes controlled flocculation, so that the agents are uniformly deposited on or in the leather fibres, which retain their felting properties despite the presence of large quantities of the coating or impregnating agent. C. O. C.

Effect of Moisture on Compressibility of Natural High Polymers (VI p. 98).
Displacement of the Iso-ionic Point of Proteins during Deformation (VI p. 98).

XIII—RUBBER; RESINS; PLASTICS

Rheology of Solutions of High Polymers. I—General Principles. K. Edelmann. *Faserforsch. und Textiltech.*, **3**, 341–344 (Sept.). **II—Structural Viscosity Measurements on Solutions of Cellulose Nitrates in Butyl Acetate.** Idem. *Ibid.*, **3**, 344–349 (Sept.). **III—Structural Viscosity Measurements on Synthetic Fibre Polymers.** Idem. *Ibid.*, **3**, 412–417 (Oct. 1952).

I—A discussion is given of the general principles of rheology, of their application to solutions of high polymers, and of the underlying molecular theory.

II—Cellulose preparations of various origins are converted into nitric esters and examined viscosimetrically in butyl acetate soln. A double-logarithmic plot is made of flow gradient against tangential force ($0–10^6$ dynes per sq. cm.) for a number of solutions of the same preparation at varying concn. The linear plot at very low concn. develops into an S-shaped curve as the concn. increases,

indicating marked structural viscosity, and the points of inflection on these curves fall on a straight line parallel to the force axis; i.e. there is a critical flow gradient that is independent of the concn. The higher the mol. wt. of the preparation, the lower the critical flow gradient. On the other hand, for different preparations at a given concn. the tangential force at the critical flow gradient is const., i.e. is independent of mol. wt. Structural viscosity measurements can therefore form the basis of an absolute method of mol. wt. determination.

III— Solutions of polyacrylonitrile preparations in dimethylformamide are examined by the procedure outlined above. They show considerable structural viscosity, and their behaviour with respect to the critical flow gradient is precisely parallel to that described for cellulose nitrate solutions. On the other hand, solutions of vinyl chloride–vinylidene chloride copolymer (in *cyclo*hexanone) and of the polycondensates from ε-aminocaproic acid and from terephthalic acid and ethylene glycol (in cresol) show only low structural viscosity and depart only slightly from Newtonian behaviour, even under the very high tangential forces used. A. E. S.

Irradiation of Linear High Polymers. K. Little. *Nature*, **170**, 1075–1076 (20 Dec. 1952).

Polymers are subjected to irradiation in the Harwell pile, and to electrons from a van de Graaff generator, and results are studied by X-ray diffraction. Bonds, some ionic but mostly covalent, are broken down; radicals produced form low-mol.wt. compounds and carboxyl end-groups in presence of oxygen, or different end-groups and low-mol.wt. compounds in absence of oxygen. They may re-form the original bonds or recombine in other ways. Factors influencing the reactions, particularly the chemical nature of the polymers such as crystallinity and presence of side-groups, are discussed. J. W. B.

Plastics— 1952 in Review. G. M. Kine. *Modern Plastics*, **30**, (5), 111–124, 187–199 (Jan. 1953).

Review of the literature (excluding patents) that appeared in 1952 and the latter part of 1951. Every aspect of the manufacture and use of plastics is covered, there being 404 references. C. O. C.

Reactions of Urea with Formaldehyde. I— Kinetics of Bishydroxymethylurea Formation. R. Květoň and M. Králová. *Chem. Listy*, **46**, 403–407 (1952): *Chem. Abs.*, **46**, 10,810 (25 Nov. 1952).

Urea condenses with HCHO stepwise in two reversible reactions. Reaction of $H_2N{\cdot}CO{\cdot}NH{\cdot}CH_2OH$ (I) with HCHO is of the second order and has an activation energy of 12·2 kg.cal.; the reverse reaction has an activation energy of 16·9 kg.cal. Formation of $CO(NH{\cdot}CH_2OH)_2$ (II) is more affected by pH in the 5–8 range than is formation of I. Equilibrium constants and heat of the reaction I + H·CHO = II (4600 kg.cal.) are given over the range 40–70°C. C. O. C.

Kinetics of the Condensation of Melamine with Formaldehyde. M. Okano and Y. Ogata. *J. Amer. Chem. Soc.*, **74**, 5728–5731 (20 Nov. 1952).

Measurement of the rates of condensation of melamine with formaldehyde in aqueous media of pH 3·0–10·6 at 35, 40, and 70°C., using both iodometric and sulphite methods, shows that the primary product of condensation, hydroxymethylmelamine, consumes iodine just like formaldehyde, and therefore the result of the iodometric method indicates the amount of formaldehyde plus that of hydroxymethyl group present, while the sulphite method estimates solely unchanged formaldehyde. Evidence has been found that formation of hydroxymethyl compounds occurs at 35–40°C. except in acidic solution, and that the reaction is reversible at all pH, its forward rate being proportional to either [Melamine] × [H·CHO], [Melamine] × [Conjugate acid of H·CHO], or [Conjugate base of melamine] × [H·CHO] according to the pH of the media. The rate of irreversible condensation of hydroxymethylmelamine with melamine in neutral and acidic medium at 70°C., where hydroxymethylation is very rapid, is determined by the condensation step of conjugate acids of hydroxymethylmelamines with melamine, the rate being expressed as [Melamine] × [H·CHO]. The rate equations based on the reaction mechanisms suggested by the above satisfy the relationship between pH and the rate constant found experimentally. C. O. C.

PATENT

Stabilisation of Vinyl Halide Polymers to Discoloration by Light. De Bataafsche Petroleum M.-S. *BP* 684,515

Vinyl halide polymers are stabilised to deterioration by light by incorporating therein 0·01–5% of an ester of a polycarboxylic acid and an unsaturated alcohol having an olefinic linkage > 4 C atoms from the carbinol C, e.g. diallyl maleate, and 0·01–5% of either an epoxy-containing compound, e.g. poly(allyl glycidyl ether), or a salt of a weak carboxylic acid, e.g. strontium naphthenate. W. G. C.

Kinetic and Thermal Study of the Polymerisation of Caprolactam (VI p. 99).
Stabilising Polythene to Sunlight (VI p. 99).
Polyvinyl Acetate and its Derivatives as a Basis for Textile Auxiliaries (X p. 104).

XIV— ANALYSIS; TESTING; APPARATUS

Possibility of Exact Control of Auxiliary Textile Products. G. Schwen. *Teintex*, **17**, 755–768 (Dec. 1952).

Methods used for the measurement of some properties of surface-active agents are described and discussed. Absolute measures of wetting power are obtained from surface tension measurements, relative values from flotation or immersion methods. The variations of wetting power with concentration of wetting agent and temperature are briefly considered, and the stabilities to hard water of three wetting agents compared. Static and dynamic methods of estimating foaming power are described, and also a method of examining the dispersing power of surface-active agents for lime soaps. Apparatus for determining the effect of wetting agents on the contraction of cotton fibres in caustic soda solution is described, and a method of estimating detergent power, using wool treated with olive oil containing Sudan BR and a laboratory washing device. The effect of increasing chain length of secondary sulphonated alcohols on wetting power and detergent and foaming actions is briefly considered. W. R. M.

Report on Sulphonated Amine Intermediates in Coal-tar Colours. Determination of Lake Red C Amine in D&C Red No. 8 and 9. N. Ettelstein. *J. Assocn. Off. Agric. Chem.*, **35**, 419–421 (1952): *Chem. Abs.*, **46**, 11,473 (25 Nov. 1953).

The results of collaborative study of a method based upon the absorption of the intermediate in slightly alkaline solution at 247 mμ. are given. C. O. C.

Report on Paper Chromatography of Coal-tar Colours. D. H. Tilden. *J. Assocn. Off. Agric. Chem.*, **35**, 423–435 (1952): *Chem. Abs.*, **46**, 11,473 (25 Nov. 1952).

Discussion of paper chromatography for separating and partly identifying some dyes. The R_f values of many certifiable dyes on paper with various solvent systems are recorded, and tables given showing the colour reactions produced in swatches of chromatograms with conc. HCl, conc. H_2SO_4, 10% NaOH, and conc. NH_4OH. C. O. C.

Studies in Coal-tar Colours. XI— D&C Red No. 30. C. Stein and K. A. Freeman. *J. Assocn. Off. Agric. Chem.*, **35**, 491–495 (1952); **XII— D&C Orange No. 3.** *Ibid.*, 495–499: *Chem. Abs.*, **46**, 11,473 (25 Nov. 1952).

Spectrophotometric data for $CHCl_3$ soln. of purified D&C Red No. 30 and aq. soln. of purified D&C Orange No. 3 are given. The dyes can be determined satisfactorily by either spectrophotometry or $TiCl_3$ titration. C. O. C.

Subsidiary Dyes in FD&C Colours. Determination of Lower Sulphonated Dyes in FD&C Yellow No. 5. M. Dolinsky. *J. Assocn. Off. Agric. Chem.*, **35**, 421–423 (1952): *Chem. Abs.*, **46**, 11,689 (25 Nov. 1952).

Report of a collaborative study of a method based upon the absorption of the dye at 434 mμ. C. O. C.

Paper Electromigration of Flavonoids and Sugars using a High Constant-voltage Current. Y. Hashimoto, I. Mori, and M. Kimura. *Nature*, **170**, 975–976 (6 Dec. 1952).

Ultraviolet Papyrography— an Automatic Analytical Apparatus using Filter Paper without a Colour Reagent. Y. Hashimoto and I. Mori. *Nature*, **170**, 1024–1025 (13 Dec. 1952).

Ultraviolet radiation within the region from 2537 A. to the visible is passed through the paper to an amplifier and then to a recorder. Four methods may be used— (i) densitometry of finished papers prepared by paper chromatography or electromigration; (ii) automatic paper chromatography; (iii) automatic paper electromigration; (iv) automatic paper electrochromatography. In method (i) the paper is scanned from start to solvent front, permitting the R_f values of substances absorbing in the ultraviolet region to be readily determined. With the other three methods, a monochromatised beam is projected on the fixed paper, and a sample is run by using a developing solvent or an electric current. Rapid automatic analysis of colourless substances results. By the techniques described, which may be qualitative or quantitative, ultraviolet papyrograms for flavonoids, alcohol xanthates, and some organic bases have been prepared. A diagram of the apparatus and a typical papyrogram are given.
J. W. D.

Determination of Titanium in Pigments and Ores— Titrimetric Method. J. A. Rahm. *Anal. Chem.*, **24**, 1832–1833 (Nov. 1952).

Material to be analysed is dissolved in 18M-H_2SO_4 containing $(NH_4)_2SO_4$ (50% wt./vol.) in an Erlenmeyer flask, and reduced at the boil with Al–HCl. Fumes are led by a simple delivery tube into $NaHCO_3$ soln., where they are trapped. After cooling to < 60°c. the contents of the flask are titrated with $FeCl_3$ in presence of KCNS. Ores and residues are initially dissolved by fusion with $KHSO_4$. When Fe is present, more reducing agent is required; Cu and Sb interfere. A small blank titration may result from Sn and Ti, which are frequent impurities in metallic Al. Despite its simplicity, this method is claimed to give accuracy superior to that of the Jones reductor method, this conclusion being based on the results of some 3000 analyses.
J. W. D.

Detection of Cinnabar in Paints and Printing Inks. M. Palumbo. *Ind. Vernice* (Milan), **6**, 89 (1952): *Chem. Abs.*, **46**, 11,704 (25 Nov. 1952).

Hot alkaline $Na_2S_2O_4$ or other alkaline reducing agent turns HgS dark grey or black by forming Hg. Cold or hot conc. HNO_3 converts the product back to bright red HgS. This is used to detect commercial HgS after removing it from the vehicle and organic pigments.
C. O. C.

Micro-methods in the Paint and Lacquer Laboratory. VII— Assay of Lithopones. VIII— Analysis of Zinc Oxide. *Ibid.*, 267–269: *Chem. Abs.*, **46**, 10,637 (10 Nov. 1952).

VII— A micro-method is given based on evaporation of an HCl soln. of the sample to remove H_2S and subsequent titration of the Zn with Na ethylenediaminetetra-acetate using Eriochrome Black T as indicator.

VIII— Tartaric acid is added to a soln. of the sample, which is then made ammoniacal. In one aliquot the sum of Pb and Zn is determined by titration with Na ethylenediaminetetra-acetate using Eriochrome Black T as indicator; KCN is added to a second aliquot, in which Pb is then titrated alone.
C. O. C.

Salting-out Chromatography of Amino Acids and Peptides in Filter Paper and other Media. L. Hagdahl and A. Tiselius. *Nature*, **170**, 799–800 (8 Nov. 1952).

Adsorption by filter paper of certain proteins is greatly increased in presence of (usually concentrated) salt solution. The principle has wide applicability in chromatography, being advantageous where lack of solubility in one phase makes application of the partition method difficult.
J. W. B.

Measurement of Colours and Shades. C. Bravo. *Chim. Peintures*, **15**, 250–255 (1952): *Chem. Abs.*, **46**, 10,701 (25 Nov. 1952).

Description of a photocolorimeter for reflection and transmission.
C. O. C.

High-sensitivity Differential Colorimeter. L. G. Glasser and D. J. Troy. *J. Optical Soc. Amer.*, **42**, 652–660 (1952): *Chem. Abs.*, **40**, 10,702 (25 Nov. 1952).

A high-precision tristimulus filter colorimeter, suitable for both research and industrial use, reproducibly measures C.I.E. tristimulus values X, Y, and Z and ratios of tristimulus values, sample : standard, to four significant figures for most colours. Colour differences considerably smaller than those a trained observer can detect visually can be measured on all colours from whites to blacks of about d.4.
C. O. C.

Fastness Tests on Textiles. C. Zuber. *Teintex*, **17**, 708–723 (Nov. 1952).

The development of methods of fastness testing and assessment of the results of tests proposed by various organisations leading up to the I.S.O. tests is discussed with special reference to fastness to washing and light.
B. K.

PATENTS

Colour Matching. General Electric Co. *USP* 2,602,368

Apparatus in which the spectrophotometric curve of a sample can be visually compared with the combined spectrophotometric curves of more than one colour element allows rapid and simple determination of the relative amounts of the colour element, e.g. dye or pigment, necessary to produce a match.
C. O. C.

Indicating or Comparing the Content of Constituents in Flock. Slumberland. *BP* 685,370

Flock is blown into a cylindrical chamber, where it encounters a controllable downward jet of air, and is driven into a lower, narrower part containing interchangeable spaced grids or sieves, of dimensions depending on the fractions it is desired to examine. This enables the starting material to be classified in terms suitable for the proposed standards for the feathers and rag flock industry.
J. W. B.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XI— Oxidation Potentials of Naphthidine, 3:3′-Dimethylnaphthidine, and their Sulphonic Acids (IV p. 90).

Practical Application of the Iodine Value Conversion Diagram in Studies of Crystallisation Processes (VI p. 98).

Spectrophotometric Analysis of Acidified Sodium Chlorite Solutions (VII p. 101).

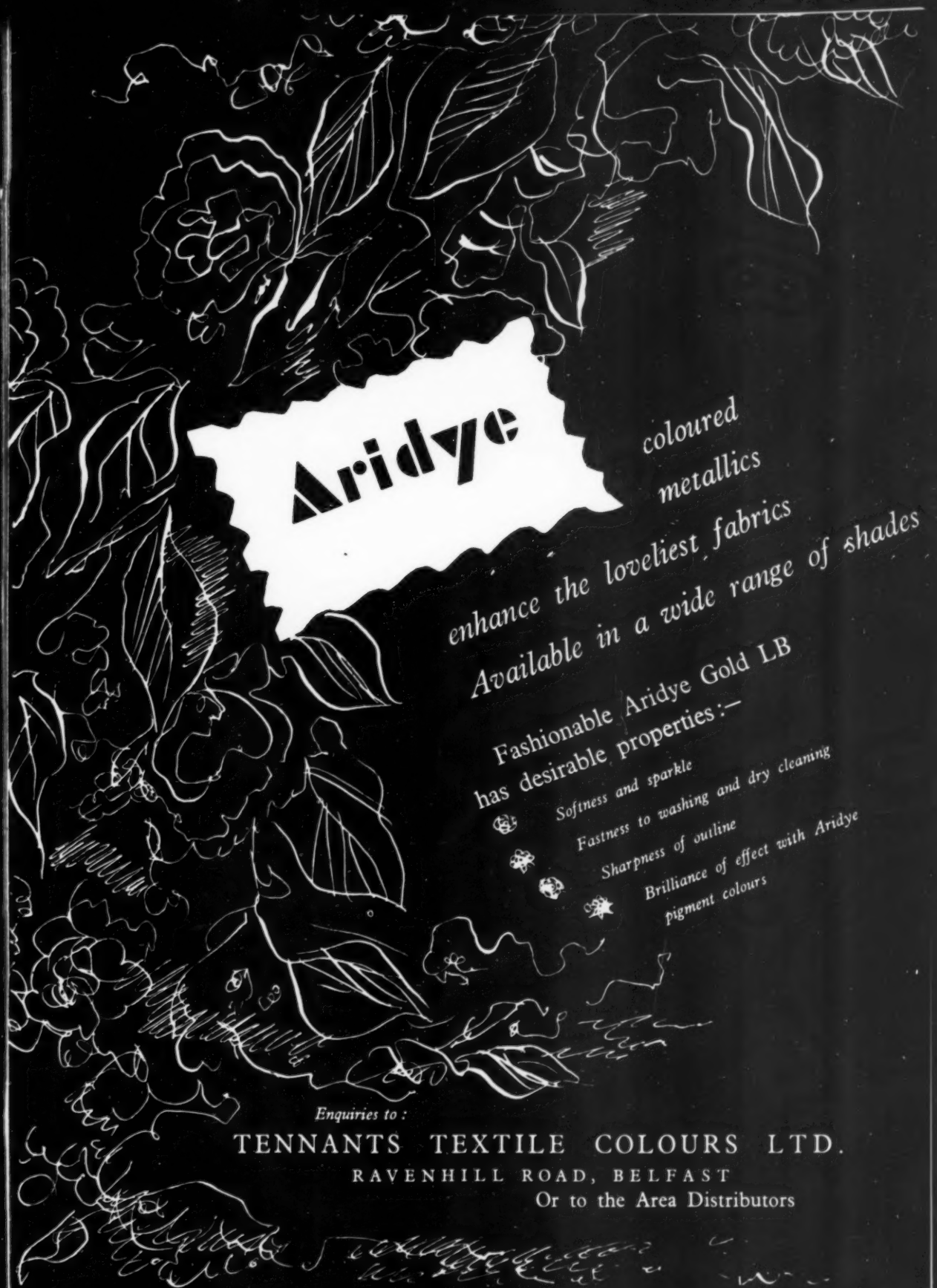
Spectrophotometry of Biuret Complexes as a Method of Investigating Proteins and Peptides. XIV— Effect of the Amino Acid Composition of a Polypeptide on the Absorption Spectrum of its Biuret Complex (XII p. 106).

Rheology of Solutions of High Polymers. I— General Principles. II— Structural Viscosity Measurements on Solutions of Cellulose Nitrates in Butyl Acetate. III— Structural Viscosity Measurements on Synthetic Fibre Polymers (XIII p. 106).

XV— MISCELLANEOUS

Objectivity of Colour Preferences. G. W. Granger. *Nature*, **170**, 778–780 (8 Nov. 1952).

Groups of subjects arrange sets of standard Munsell colours in order of preference, and adjust the areas of pairs of colours to obtain the most pleasing balance. Results are discussed in terms of colour-selection theories, and the conclusion is reached that preferences are objective in the sense that they are considerably independent of personal taste, and are dependent to some degree on inherent stimulus properties.
J. W. B.



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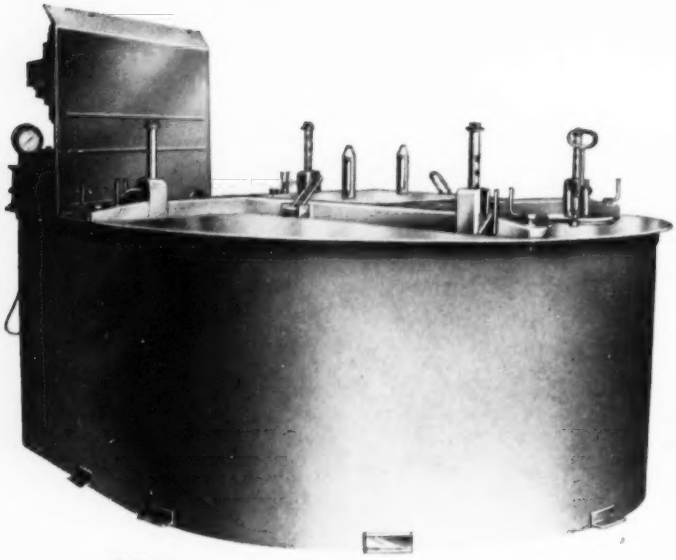

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The Textile Institute and the Society of Dyers and Colourists have jointly published a Review of Textile Progress, Volume 3 relating particularly to the year 1951. There are 32 contributors in the Review and the authors are experts in the various sections for which they are responsible

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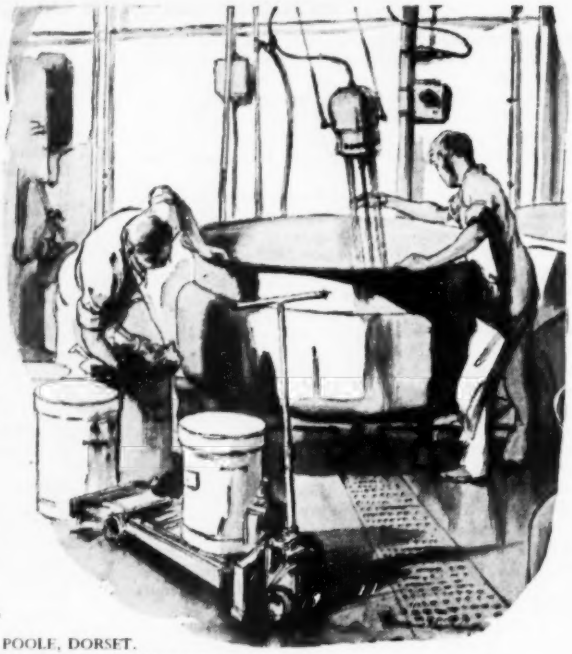

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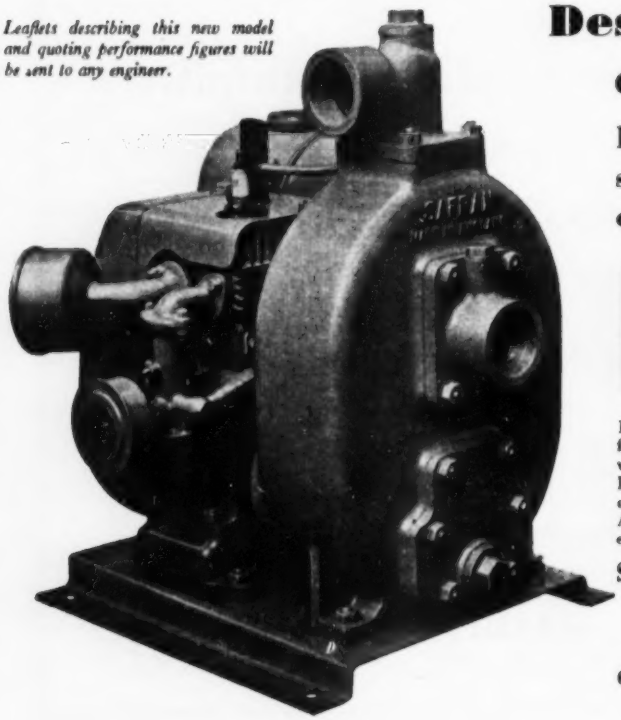

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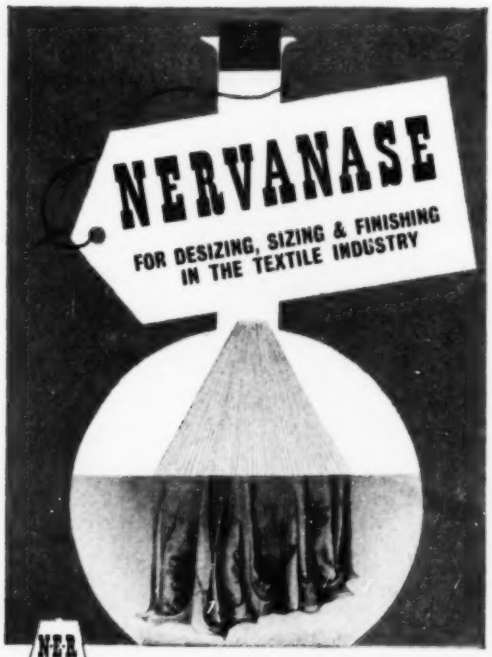

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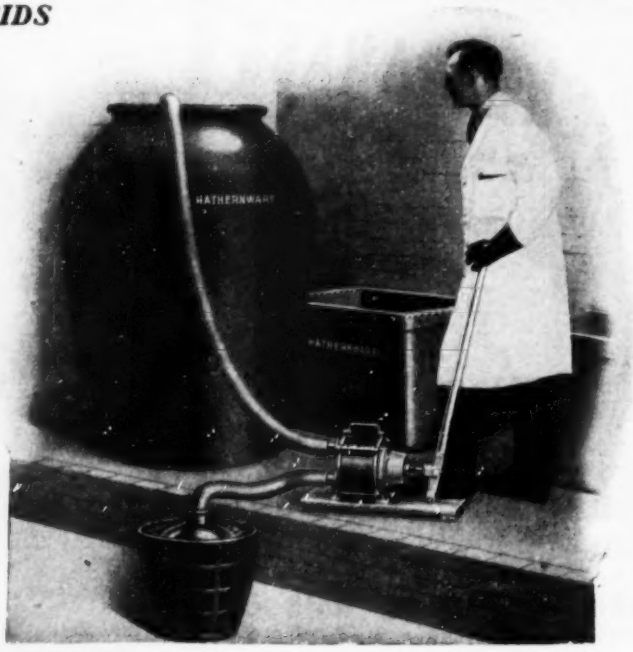

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